#### PATENT ABSTRACTS OF JAPAN

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#### (54) INTERMEDIATE FILM FOR LAMINATED GLASS AND LAMINATED GLASS

#### (57)Abstract:

PROBLEM TO BE SOLVED: To obtain a laminated glass which maintains basic performance necessary for a laminated glass and little efflorescence in the peripheral part even in a high humidity atmosphere by forming an intermediate film comprising a plasticized polyvinyl acetal resin film having a specified or lower haze when the film is immersed in water.

SOLUTION: When the intermediate film for a laminated glass comprising a plasticized polyvinylacetal resin film having 0.3 to 0.8 mm thickness is immersed in water at 23° C, efflorescence of the film after 24 hours is controlled to ≤50 % haze. To obtain the haze value above described, sodium safts and potassium salts which cause the efflorescence and which are mixed from the source material are controlled to have ≤10 µm particle size and to give ≤50 ppm sodium concn. and ≤100 ppm potassium concn. in the intermediate film. To effectively prevent efflorescence at high humidity, compds, which can form complexes with sodium salts and potassium salts, and org, acids and amines compatible with the resin and the plasticizer are preferably added as dispersants.

11-043152A [OLAIMS]

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#### AIMS

sim(s)]

aim 1]An interlayer for glass laminates when it is an interlayer for glass laminates which consists slassicozod polyvinyi polyvinylacetal resin films and said 0.3–0.8-mm-thick interlayer is immersed in \*\* water, wherein Hayes of 24 hours after is 50% or less.

sim 2]The interiayer for glass laminates according to claim 1 whose particle dismeter of sodium in an interlayer is 10 micrometers or less.

aim 3]The interlayer for glass laminates eccording to claim 1 or 2 whose particle diameter of fum salt in an interlayer is 5 micrometers or less.

aim 4] The interlayer for glass laminates according to claim 1, 2, or 3 whose sodium concentration in interlayer is 50 ppm or less, sim 5]The interlayer for glass laminates according to claim 1 whose particle dismeter of potassium ; in an interlayer is 10 micrometers or less. sin B1The interlayer for glass laminates of alm 81The interlayer for glass laminates according to claim 1 or 5 whose particle diameter of

assium salt in an interlayer is 5 micrometers or less.

aim 7]The interlayer for glass laminates according to claim 1, 5, or 8 whose potassium

scentration in an interlayer is 100 ppm or lass.

aim 8]The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 8, or 7 which is a thing theiring a compound which can form sodium salt and potassium salt, and a complex.

taining amine which is compatible in organic acid which is compatible in resin and a plasticizer, aim 9]The interlayer for glass laminates eccording to claim 1, 2, 3, 4, 5, 6, or 7 which is a thing

aim 10]The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 6, 7, 8, or 9 which is a in, and a plasticizer.

ng containing at least one sort chosen from a group which consists of alkali metal salt and alkaline h a particle diameter of 3 micrometers or less and whose alkaline earth metal salt is a thing with a sim 11]The interlayer for glass laminates according to claim 10 whose alkali metal salt is a thing th metal salt

all metal salt of organic acid of the carbon numbers 5–16 and whose alkaline earth metal salt is the aim 12]The interlayer for glass laminates according to claim 10 or 11 whose alkali metal salt is the tine earth metal sait of organic acid of the carbon numbers 5~16. ticle diamater of 3 micrometers or less.

aim 13]A glass laminate characterized by making the interlayer for glass laminates according to m 1, 2, 3, 4, 5, 8, 7, 8, 8, 10, 11, or 12 came to intervene between glass of a couple at least.

anstation done.]

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### MAILED DESCRIPTION

stalled Description of the Invention]

<u>...</u>

id of the Invention]This invention relates to the giass laminate which used the interlayer for glass inakes, and the above-mentioned interlayer for glass laminates, and the above-mentioned interlayer for glass laminates.

as so that it may fell within a proper range. Namely, if the danger of the glass fragment damaged by nan body etc. If the adhesive strength of an interlayer and glass is too weak becomes high and the ulteneously damaged by the shock from the outside, etc., and the danger of the exhission fragment wide and happens, the impact-absorbing effect and a penetration preventive effect become large. ic performance is good and excellent in safety. That is, by the pariphery of a glass laminate, when riayer which consists of a piesticization polyvinyl butyral between the glass plates of at least two above-mentioned glass faminate is placed into a humid atmosphere, since the interlayer touches an interlayer extends arises at the same time glass is damaged while glass breakage reaches far oribed below is participating in this albinism. In order to demonstrate the function as the above ntioned glass (ammate enough, it is required to adjust the adhesive strength of an interlayer and ts. Since the phenomenon in which partial interfacial peeling of an interlayer and glass happens, shook from the outside, etc. separating and dispersing from an interlayar, and deing injury to a ets. It has basic performance transparency, weatherability, and an adhesive property are good, 03]This kind of glass laminate is inferior in moisture resistance, although the above-mentioned 05,00s the other hand, when there is adhesive strength of an interlayer and glass within proper id penetration resistance moreover and required for the glass faminate of a glass fragment net scription of the Prior ArtiThe glass laminate with which it comes conventionally to fasten the lesive strength of an interlayer and glass is too strong conversely. Glass and an interlayer are 04]The additive agent for performing adheaive strength adjustment of an interlayer and glass ßass and an interlay⊌r dispersing and doing an obstaele to a human body etc, becom∉s high. persing easily, for example, is widely used for the windowpane of a cer or a building. fronmental air directly, the problem which the interlayer of a periphery milks anses.

OBJTherefore, in case of the accident of transport—airplane machines, such as a car, th order to ord a shock in case a driver end a peasenger collide to glass or to prevent penetrating, in case of accident of a building, in order to prevent the missile from the outside from penetrating glass or prevent scattering of a glass fragment, it is required to sijust the adhesive strength of an interlayer and glass so that it may fall within a range proper like \*\*\*\*\*\*

O'The order to adjust the adhesive strength of an interlayer and glass within proper limits wontionally in view of the above, the adhesive strength regulator for interlayers has been mined variously. In 40-46-42703, the interlayer for glass laminates which consists of a polyviny-tal resin composition which carries out specific amount antent of 0.2 to 6.8 % of the weight of sixture and the metal alixy carries out specific as an adhesive strength regulator is proposed. The riskure and the metal alixy carries to be distribution quantity of metal alixy carries to changing the distribution quantity of metal alixy carriers an interlayer is an alwayer layer part and an interlayer man layer part, or changing the moisture content in en

08]However, the interlayer containing metal alkyl carboxylate like the above-mentioned proposal,

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## JP,11-343152,A [DETAILED DESCRIPTION]

Since the interlayer is carrying out direct contact to air in the periphery of the glass laminate if the glass laminate which moisture resistance fell and was manufactured using this interlayer is neglected under a frumid atmosphere. While the quantity of metal alky carboxylate increases, there is a problem that the albinism by moisture absorption of an interlayer happens violently. Although it can prevent whether the albinism of the above-mentioned interlayer reduces the quantity of metal alky carboxylate as much as possible, and by losing, in that case, the fatal problem as a glass laminate of the adverse strength of an interlayer and glass becoming stronger than the proper range too much, becoming easy to penetrate had occurred.

[0093]h JP.44-32185;B. contain 0.1 to 0.8% of maisture, and The manacarboxylis acid of six to 22 carbon atom, The dicarboxylis acid of four to 12 carbon atom, and the alphatic series manacarino manocarboxylis acid of two to 6 carbon atom. The interlayer for glass laminetes which cerries out 0.01-3 weight-section content of the allphatic series monoamins dicarboxylic soid of four to 5 carbon atom and at least one organic acid chosen from citrate and these mixtures per resin 100 weight acction and which consists of shaping polywhyf-acetal resin is propased.

[ROII] However, when carhoxylio acid is added, there is a problem that adhesive strength changes with time. The problem that the heat resistance and the weathersbillty of an interlayer fall under the influence of soid arises.

[0011] in the glass which pasted the glass of at least two shaets together with the plasticized—polyvinyi-acetal-resin penstituent in UP.48-5772,B. The glass laminate making sodium metal saft of the alignatic carboxylic acid of the carbor numbers 10-22 contain in this plasticized-polyvinyi-acetal-resin constituent is proposed.

1001 23in JP,53-18267,B, using the alkali metal salt or slikaline earth metal salt of monocarboxylic acid carboxylic acid is proposed as an achesive strongth regulator in the piesticized-polyvinyl-acetal-resin interlayer of a glass laminate.

[0013]In the two above—mentioned proposals, each uses metal sait of carboxylic acid with a comparatively large carbon number as an adhesive strength regulator from it being easy to dissolve in the plasticizer contained in an interlayer.

100 idjilowever, when metal salt of carboxylic acid with a large carbon number is used as an adhesive attempth regulator, there is a problem that the adhesive strength of an interlayer and glass changes with time progress (temporality). That is, even if early adhesive strength is proper, adhesive strength declines gradually with temporality, and when shocked, glass exfoliates easily, in order to prevent this adhesive strength fall, it is nocessary to keep an interlayer for one to two months under 40-50 \*\* adhesive strength fall, it is nocessary to keep an interlayer for one to two months under 40-50 \*\* keeping it under the above atmosphere for a long period of time rines difficult actually, although the fall of adhesive strength with the passage of time can be controlled, it cannot be made for there to be nothing, but the above-mentioned problem will still remain.

10015]in 4P,80-210551,A, to plesticized-polyvinyl-acetel-resin 160 weight section. The glass laminate with which the glass of at least two sheets sticks, and it comes to under it by whether 0.02 to petassium monocarboxylate 0.49 weight section and 0.01 to modified silicone oil 0.26 weight section whose carbon numbers are 1-6 contain, and the interlayer to which it acheres is indicated. However, since this metal salt solidified in particle state in the interlayer depending on the kind of metal salt used and this glass laminate caused a white blush mark, the perfect thing was not able to say from a viewpoint of preverting the white blush mark by long-term moisture absorption.

[00:16]in JP.2-41547,B, the polyvinyl-butyral sheet which uses alkali or alkeline-earth-metals formate for an adhesive strength regulator is proposed. In the Patent Publication Heisei No. 502594 [ six to ] gazette, the interlayer which added potassium acetate as an adhesive strength regulator in the

[00:1] In the three above-mentioned proposals, in order to canoel said problem in the case of using metal saft of carboxylic acid with a large carbon number as an adhesive strength regulator, metal salt of carboxylic acid with a comparatively small carbon number is used

10018]However, if metal sait of carboxylic gold with a small carbon number is used as an adhesive strength regulator. Although the problem of an adhesive strength fall with the passage of time of an interlayer and glass is canceled, the moisture resistance of an interlayer becomes incufficient and, as a result, another problem of becoming easy to start the albinism by moisture absorption to the edge

t (end) of a gess leminate occurs.

he weight. At this time, water gathers for the circumference of metal salt of carboxylic acid with a a small carbon number or its salt is decreased in order to reduce albinism, the adhesive strength (9) That is, since tygroscopicity is under the usual atmosphere (furnidity), when processing it into example so that water content may be about 0.5 or less % of the weight under the stmosphere of ridity etmosphore, an interlayor absorbs moisture and water content usually rises to about 2 to 3% iss laminate, it is common [ an interlayer ] to process it by controlling the humidity and doubling. its as a minute crystal in an interlayer, and abinism is ceused. If the addition of carboxylic acid in interlayer and glass will deviate from the proper range, and will become insufficient { impactaii carbon number of potassium acetate, magnesium scetate or potassium formate etc. which RH. Hawever, since the edge part of a giass laminate is in an unreserved state, under a high orption nature, penetration resistence, etc. of a glass laminate ].

in JP,5-188250,A. The interlayer for glass laminates currently formed from polyvinyf-acetal resin. plasticizer, and the resin composition in which alighatic series mone~ \*\* whose carbon number is 20]As a trial to improve, the white blush mark of the interlayer containing carboxylic acid metal or less contains the alkell or alkaline earth metal salt, and organic soid of dicarboxylio soid is posed.

taining polyvinyt-acetal resin, a plasticizer, carboxylic acid metal salt, and straight-chain latty acid 21] The interdayer for glass faminates which becomes JP, 7-41340, A from the resin composition reposed.

istance test is reduced. And when it is going to reduce a white blush mark further and the content 22]However, the glass taninate using the interlayer for glass laminates of the above-mentioned straight-chain fatty acid is increased, and a glass laminate is placed under relatively high posal is not still anough, although the white blush mark of the edge part after a humidity sperature, there is a possibility of producing feaming and discoloration,

esive strength regulator, the white blush mark by maisture absorption produces it. It became clear our fatest research that the impurity in the resin described below is involving as one of the cause 23]Aithaugh the interlayer of the above-mentioned proposal tries solution of a white blush mark improvement of an achesive strength regulator, also in the interlayer which is not adding the

er. Sodium saft may remain also to polyviny! afcohol and this sodium saft may cause a white blush 24]Aithough the interlayer for glass laminates of this invention uses polyviny—acetal resin as the viayer for glass laminates obtained in order to promote condensation of water, when it becomes ce the solution of sodium salt, such as sodium hydroxide and sodium bicarbonate, is used in this itralization process, for example, When those sodium salt is used superflucusly and new sodium n ingredients, the neutralization process is included when manufacturing polyvinyl-acetal resin. ticle state at the time of a polymerization and desircation and polyvinyl-acetal resin absorbs s sodium sait that remained causes [ big ] a white blush mark by moisture absorption of the , generates by neutralization, sodium sait remains in the polyviny-apatal resin obtained. rk by moisture absorption of the interlayer for glass laminates.

dings of a par prospers, in these uses, also when using the periphery of a glass laminate in the eaerved state, it is increasing, and the request to eibinism prevention is increasingly stronger. 25]m recent years, the motion which uses a glass laminste for side glasses and the various

as faminate edge part in providing the interlayer for [ little ] glass laminates, and the glass laminate istance, even when mareover placed into a humid atmosphere, there is a white biush mark of a ationed problem and is made into the purpose, Without spoiling basic performance required for is laminates, such as transparency, reatherability, an adhesive property, and penetration obsem(s) to be Salved by the sevention)The place which this invention solves the above-

sane for Solving the Problem This invention is an interlayer for giass faminates which consists of sticized palyvinyl polyvinylacetal resin films, and when the 0.3-0.0-mnr-thick above-mentioned stayer is immersed in 23 \*\* water. Hayes of 24 hours after is an interlayer for gisss laminates on is 50% or less. This invention is explained in full detail below. 27/www4.indl.innt.go\_ip/cgi-bin/tran.web\_ngi\_ajje?atw\_5+ttp%3A%2F%2Fwww4.indli... 2009/06/15

JP,11-343152,A [DETAILED DESCRIPTION]

<u> 10028]When an interlayer for glass laminates of this invention immerses a 0.3–0.8–mm-thick interlayer</u> in 23 \*\* water, Hayes of 24 hours after is 50% or less. :0029]When this invention persons immerse a 0.3–0.8-mm-thick interlayer in 23 \*\* water, Even when ieminates is humid, there were few white blush marks of a glass laminate edge part, they found out Hayes of 24 hours after was placed into atmosphere where 50% or less of interlayer for glass excelling in moisture resistance, and completed this invention.

(0030)Since it becomes insufficient to prevent a white blush mark under high humidity and it is mentioned ranga. Above-mentioned Hayes as used herein shall mean a value which used and measured an integraí equation turbidity motar 24 hours efterward, when e 0.3-0.8-mm-fhiok inferior to maisture resistance when above-mentioned Hayes exceeds 50%, it is limited to a interleyer is immersed in 23 \*\* water:

10031] An interlayer for glass leminates of this invention consists of a plasticity polyvinyl-acetal resin layer, and the above-mentioned plasticity polyvinyl-acetal resin layer uses polyvinyl-acetal resin as the main ingredients,

[0032] As the above-mentioned polyvinyl-acetal resin, average degree-of-acetalizations of a thing of mixing a plasticizer of a complement to reservation of penetration resistance --- hard --- \*\* --- there obtained will fall, in order to obtain resin, prolonged reaction time is required, and it is not sometimes is a case. If 75-mol % is exceeded, while a mechanical strength of an interlayer for glass laminates manufacture of resin if 30-mol % is exceeded, it becomes difficult to manufecture. Preferably, it is [0033]In the stove-mentioned plasticized polyvinyl acetal resin, the following [ 30 ms] % ] have a 40-75 moi is preferred, compatibility with a plesticizer falling that it is less than [ 40 mol % ], and hygrosoopioity becemes high, a white blush mark will hanpen easily. It is 64-71-mof % preferably. preferred vinyl acetate component. Since it will become easy to cause blocking at the time of desirable on a process. It is 60–75-mo! % more preferably. If it is less than [ 60 mol % ], since less than 19 mol %.

viry! stochol ingredient, and a viny! acetate component, and each of these component amounts. It can measure based on for example, a JiS K 6728" polyvinyl-butyral test method" and a nuclear magnetic [0034]The above-mentioned plasticized polyvinyl acetal resin comprises a vinyl-acetal ingredient, a resonance method (NMR).

remaining vinyf-acetal component amounts can be computed by deducting both the zbove-mentioned aicohol component ameunt and the amount of vinyl acetate components can be messured, and the [0035]When the above-mentioned polyviny-acetal resin is except polyvinyl butyral resin, a vinyl component amounts from 100.

Subsequently, reaction temperature is raised to 70 \*\*, it ripes, a reaction is completed, and a method [0036]The above-mentioned polyvinyt-apetal resin can be conventionally manufactured by a publicly of performing neutralization, rinsing, and desiocetion and obtaining pewder of polyvinyf-acetal resin, scetalization reactions are advanced, adding and etirring a necessary acid catalyst and aldohyde. known method, for example, solution obtained by dissolving palyvinyl alcohol in warm water --- a predetermined temperature --- for example, it holds at 10-20 \*\* preferably, and 0-95 \*\* of etc. are mentioned after that.

(8038)Since it is preferred to set a vinyl acetate component of polyvinyl-acetal resin obtained to less preferred thing beyond 70 mal %. When it is less than [ 70 mal % ], the transparency of resin and heat 19037]As polyvinyl alcohol used as the above-mentioned raw material, a thing of the average degrees of polymerization 500–5000 is preferred, and a thing of the average degrees of polymentation 1000– abtsined may fall. If 5000 is exceeded, shaping of a resin layer will parry out -- hard --- \*\* --- there than 30 mof % therefore, a saponification degree of the above-mentioned polyvinyl elechol has a [0039]An average cogree of polymerization and a saponification degree of the above-mentioned 2500 is more preferred. When it is less than 500, the penetration resistance of a glass laminate resistance may fell and reactivity may also talk it is a thing beyond 95 mol % more prefereby. are things and, moreover, intensity of a resin layer may become strong too much.

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As the above-mentioned aldehyde, aldohyde of the carbon numbers 3–10 is preferred. The moldability

exceeded, the reactivity of acetalization will fall, and moreover it will become easy to generate a

of resin layers with a sarbon number enough by less than three may not be obtained. If 10 is

block of resin during a reaction, and will become sasy to follow difficulty on composition of resin.

pokyinyi alcohol can be measured, for example based on JIS K 6726" polyvinyi alcohol test-method."

40]kt is not limited especially as the above-mentioned sidehyde, but For example, propionaldehyde, izaldehyda, and a cinnamaldahyda, aromatic series, alicycle feilews aldehyde, etc. are mentioned utylelčehyde, isobutyraldehyde, a valeraldehyde, Aliphatic series, such as n~hexylaldehyde, 2~ rag, and ri-buty/sidehyde of the carbon number 4 is excellent also in weatherability by use of winyt-abetal resin obtained and moreover becomes easy [ manufacture of resin ], it is more ylbutylaidchyde, and n-ootyl aldchyde. Since adhesive strength of each rasin layer becomes ylbutylaidehyda, n~hestylaidehyda, n~ootyl aldahyda, n~nonyl aldahyda, n~dacyl aldahyda, farably, they are n-butylaidehyde of the carbon numbers 4-8, n-hexylaidahyde, 2farred. These may be used alone and two or more sorts may be used together.

neter of potassium salt in an interlayer, it is preferred that it is 10 micrometers or less, and it is 5 rometers er lass more preferably. The finer moderate still more desirable one of particle diameter prayer is 10 migrometers or less, and it is 5 micrometers or feed more preferably. About particle 41]In an interlayer of this invention. It is preferred that perticle diameter of sodum saft in an

42]Since condensation of water may be promoted when particle diameter of the above-mentioned lium seit exoeeds 10 miorometers or particle diameter of potassium salt exceeds 10 miorometers, particle diameter of potassium sait, also in polyvinyf-acetal resin, since perticle diameter may be assium salt may decrease in process of film production, As for particle diameter of sedium salt an become a big cause of a white blush mark by moisture absorption of an interlayer obtained. assium sait point out particle diameter in inside of an interlayer, Although particle diameter of 43]Aithough particle diameter of the above–mentioned sodium saft and particle diameter of lium salt in polyvinyl-scetal resin which is the main raw material, and particle diameter of d, it is preferred that it is in a mentioned range.

44]Particle diameter of sodium sait in the above-mentioned interlayer and potassium sait can be asured by imaging of a secondary ion image which used a time-of-flight type secondary-ionss-spectroscopy device (TOF-SIMS).

ferably, sodium concentration is 0.5 ppm or mare 15 ppm or less, and patassium concentration is 45]In an interlayer of this invention, it is preferred that sodium concentration is 50 ppm or less. aut patassium concentration in an interlayer, it is preferred that it is 100 ppm or less. More ppm or more 100 ppm or less.

rdeyer exceeds 50 ppm and potassium centents exceed 100 ppm, a white blush mark may become lium element and a potassium element is visualized if a sodium content in the above-mentioned ning to be used, such as water and raw material, by preparation of resin may be needed, great 46]Since it will grow up to be even a size by which a water molecule which gathered around a tarkable. Preparing an interlayer less than 0.5 pom in any { of a sedium content in the abovertioned interlayer, and potassium contents I case, Treatment which lengthens dramatically a cess of washing a residual sodium element or a potassium element, or raises the degrees of e and expense may be required, and it may not be desirable practically.

47]Sodium concentration and potassium concentration in the above-mentioned interleyer can be sne of a decomposition product with ultrapure water, it is the method of quantifying by the IOPntified by ICP luminescence ultimate analysis. After the above-mentioned ICP luminescenae meta analysis heats and decomposes a sample with suffuric soid and natic acid and sets the

redation of resin itself in a generation reaction of polyvinyl-acetel resin which is a praecss before cass ean prevent an acid catalyst like indispensable chloride (HCI) remaining in resin, and causing 48] Mixing of the above-mentioned sodium and/or potassium. For example, in order to neutralize acid catalyst of suffuric acid used for a reaction, chloride, etc. in preparation of polyvinyl-acetal 49]In a manufacturing method of the above-mentioned polyvinyi-acetal resin, a neutralization in, it originates in having used a neutralizer having contained a sodium element or potassium nents, such as sodium carbonate, sodium bioarbonate, sodium acetate, sodium hydroxide, assium carbonate, potassium bicarbonate, potassium acetate, and a potassium hydrate.

50]As the above-mentioned neutralizar, alkali metal sait and alkaline carth metal sait can be used. in if alkaline-earth metals remain so much in an interlayer unlike an alkaline metal, they are ferred at a point which ean control a white bluch mark under high humidity. x//wwwd.ipdl.mpit.go.jp/ogj-bis/trsn.web.ogi\_gje?atw.jr?http%3A%2F%2Fwww4.ipdli... 2009/06/15

JP,11-343152.A [DETAILED DESCRIPTION]

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0053]For example, an alkaline metal contained in the above-mentioned pure water can be reduced to 100511As the abovermentioned alkeline earth metal sait, caloium sait, such as barium sait, caloium potassium salt, such as carboxylic acid and octylic acid, etc. as an adhesiye strength regulator, a sodium element or a potassium element contains in used water, raw matsrial, especielly polyvinyl hydroxide, such as magnesium saft barium hydroxide, such as aerated water matter magnesum. I ppm or less by using ion exchange water. On the other hand, an alkalina metal contained in [0052] Mixing of the above-mentioned sodium and/or potassium, When adding sodium saft or magnesium hydroxide, and basic magnesium carbonate, etc. ere mentioned, for example airohol, etc., and it happens also by a case where these remain in an interlayer.

or less % of the weight, sodium elements with difficult washing contained in resin can be reduced, it is 10055]In a manufacturing mathod of the above-mentioned polyvinyl-acetal resin, it can also carry out 10054]Therefore, when content of sodium acetate uses a polyvinyl alcohol raw material which is 0.4 stabilized by washing strengthening etc., and a sodium element can be 50 ppm or less.

polyvinyi alouhol originates is sodium ecetate generated in a manufacturing process of a polyvinyi

aconol ran material when saponifying polyvinyl acetate, and content is usually 0.4 to 1.5 % of the

by thisting polyvinyl-acetal resin, without performing the above-mentioned neutralization process, until to a white blush mark of a resin layer obtained can be held down to below the specified quantity until remains of an acid catalyst by [ of 60 \*\* or less ] drying at fow temperature comparatively, corrosion it becomes pil five or more, and also drying below 60 \*\*. By fully rhishing, alkali metal content leading it becomes pH five or more. While preventing resin deterioration by mixing of an alkaline metal, and by acid of a dryar can be prevented. Although a conventional method may be sufficient as a drying method, especially its vectum-drying method is efficient, and excellent.

deterioration by mixing of an alkaline metal, or remains of an soid catalyst. At the time of washing, by efficiency by not less then 40 \*\*. If wash water is lower than 40 \*\*, resin will not fully swell but it will using 40-60 \*\* wash water preferably, resin in a stury swells, and acid (HCl) incorporated into resin water. It is fur reising washing efficiency by temperature of water used at the time of washing being he hard to go up an effect. When wash water is more expensive than 60 \*\*, while softening of resin not less than 40 \*\* paying attention to resin in a slurry swelling above 40 \*\*, and preventing resin (0056)In the above-mentioned washing process, it is preferred to wash with not less than 40 \*\* and its neutralized substance (alkaline metal inclusion) are flushed easily, and can raise washing may take place, particles may coalesce, formation of a blook may be seen and a thing of stable particle dismeter may not he obtained, improvement in a large affect cannot be expected as compared with 60 \*\* water, but it becomes useless also in energy.

chloride remover of a reaction using epoxide, and a method of performing fim formation can also be compounding polyvinyr-acetai resin, Polyvinyr-acetai resin can be obtained as a stop agent and a mentioned after that it is expressed with following general formula (I) as the above-mentioned 10057]In order to prevent mixing of the above-mentioned sodium and potessium, carry out the acetalization reaction of polyving sloohul, a chloride catalyst, and the aldahyde, and it faces epoxíde, [0058]

Formula 1]

(:)R' -- CH-(CH2), -- CH----R'

"3"-epoxide, auch as trimethylene oxide, e tetrahydrofurar, and tetrahydropyran, 1,5"-epoxide besides 1,2-spoxide, etc. are mentioned — these — sne sort — or two or more sorts may be used together. 0058)(R' and R' express hydrogen or an alkyl group.) n expresses the integer of 0-3 1,4-epoxide, Especially as epoxide, othyleneoxide, propylene cxide, etc. are preferred.

mentioned epoxide with the neutralizer of a chloride catalyst, and stopping an acctalization reaction 10660]As amount of the above-mentioned enoxide used, the effective doss which can perform stop of a reaction and chioride removal can be used. Degradation of resin by mixing of an alkaline metal and remains of an acid catalyst can be prevented by replacing the method of using the aboveusing epaxide, and also removing chloride.

0061]In this invention, as for an interlayer for glass laminates, in order to prevent a white blush mark

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er high humidity more effectively, it is preferred that it is what adds a dispersing agent. By adding olyvinyl-acetal resin and a plasticizer can be distributed, and particle diameter of these elements above-mentioned dispersing agent, a sodium compound, a potasslum compound, eta, which exist be made small.

spound which can form sodium salt and potassiom sait, end a complex, resin, and a plasticizer as 82] Amiriz which is compatible in organic acid, resin, and a plasticizer which are compatible in a above~mentioned dispersing agent is mentioned.

83] The compound which can form the above-mentioned sodium saft and putassium sait, and a we-mentioned palywhyt-acetal resin absorbs moisture, in order to make it hard to approach in plex can control a white blush mark of an interlayer for giass faminates obtained, even if the er by carrying out hydrophobing of the circumference of metal salt, such as sodium salt and

assium salt, and a complex. For example, effrylenediaminetebracetic acid, salicylaldehyde, salicylid othylgiyoxime, 1.1–cyclohexane 2 acetic acid, salicylaidaxime, a glycine, etc. are mentioned. These 64]It is not limited especially as a compound which can form the above-mentioned codium and 1, Salicylanilide, oxalic acid, a 1,10-phananthroline, an acetylacetone, 8-frydroxyquinoline, be used alone and may use two or more sorts together.

in, it is preferred that they are 0.02 - the amount part of duplexs to the polyvinyf-zoetal resin 100 ve-montioned weight section. If a proventive offect of a white blush merk by moisture absorption assium salt, and a complex is based on quantity of metal salt which remains to pelyvinyl-acetal eeded, compatibility with the above-mentioned polyvinyl-acatal resin may become poor, and a s become insufficient if it is less than 0.02 weight sections and the amount part of duplexs is 65]Athrough an addition of a compound which can form the above-mentioned sodium and blem may arise in transparancy. It is 0,05 to 1 weight section more preferably.

88]As the above-mentioned dispersing agent, amine which is compatible in organic acid, resin, and lasticizer which are compatible in resin and a plasticizar can also be used. As organic acid which is 67]R is using at least one sort more preferably from a group which consists of suffonic acid whose spatible in the above-mentioned resin and a plasticizer, polymer acid, such as moromer acid, such suffenie ació, carboxylia ació, phospharic ació, and nitrie ació, polysuffene ació, and palycarboxylic bon numbers are 2-21, carboxylic sold whose carbon numbers are 2-20, and phasphoric acid ete, are mentioned, and sulfonic acid, carboxylic acid, and phosphoric acid are especially ressed with following general formula (II) as organic acid which is compatible in the aboveferred. These may be used independently and may be used together two or more sorts. thaned resin and a plasticizer.

rophobicity, compatibility with polyvinyl-acetsi resin wersens, and there is a pessibility of causing nbers are 1–18, or the aromatic system hydrocarbon group whose carbon numbers are 1–18.)  $\mathbb{R}^4$  $69\%\mathrm{R}^3$  expresses among a formula the aliphatic series system hydrocarbon group whose carbon 78In the suifonic soid whose shove-mentioned carbon numbers are 2-21, if a carbon number is resses a hydrogen atom, the aliphetic series system hydrocarbon group whose cerboti numbers than two. When hydrophilic nature may become high, compatibility with polyvinyl-acetal resin versen, distributing may become insufficient and a carbon number exceeds 21, it becomes 1—18, or the aromatic system hydrocardon group whose carbon numbers are 1—18, se separation. A carbon number is a thing of 7-18 more preferably.

hatic series system, the thing of an aromatic system, etc. The above-mentioned carbon number is limited especially as sulfonic acid which is 2--21. For example, benzenesulfonic acid, naphthaiene 11)As sulfanic acid which is 2-21, the abave-mentioned carbon number can use the thing of an onic acid, alkyi sulfenio soid whose carbon numbers of an alkyl group are 2~21. The carbon ther of an alkyl group is alkyl naphthalene sufforio acid etc. which are 2-11, and the carbon ber of the alkylbenzene sulfanic acid which is 2-15, and an alkyl group specifically, P- 11/www4.jpdlinpit.go,jp/cgj-bin/van.web.cgi.ejjelatw.u=http%3A%2F%2Fwww4.jpdli... 2009/06/15

# JP,11-343152,A [DETAILED DESCRIPTION]

acid, mestylene auffonic acid, etc. are mentioned. These may be used sione and may use two or more toluenesulfonio acid, dodecylbenzenesulfonio acid, camphor sulfonio acid, hydroxypropanesulfonio

part of duplexs, degradation of resin may be promoted or this suffenic soid itself may cause a white mentioned weight section. If a preventive effect of a white blush mark according that they are less than 0.01 weight sections to moisture absorption may become insufficient and exceeds the amount preferred that they are 0.01: - the amount part of duplexs to the polyvinyl-acetal resin 100 above-10072]As for an addition of sulfonic acid whose above-mentioned carbon numbers are 2-21, it is blush mark. It is 0.03 to I weight section more preferably.

hydrophabidity, oempatibility with polyvinyl-acetal rasin worsens, and there is a possibility of causing 10073]in carboxyiic acid of 2-20 the sbove-mentioned carbon number, When hydrophilic nature may become it high that a carbon number is less than two, compatibility with polyvinyr-acetal resin may worsen, distributing may become insufficient and a carbon number exceeds 20, it becomes phase separation. A carbon number is a thing of 6-14 more preferably.

acid, pimelio scid, sebacic acid, cloic acid, benzole scid, tollic acid, naphthoia scid, 1,1-cyclohexane 2 (0074] As carboxylic acid which is 2-20, the above-mentioned carbon number can use a thing of an ethylbexyl acid, lauric acid, myristic acid, stearic acid, oxalic acid, makoric acid, succinic acid, adiple aliphatio series system, a thing of an aromatic system, etc. It, may be dicarboxy;ic acld, The above acetic ecid. salicyfic acid, etc. are mentioned. These may be used alone and may use two or more mentioned carbon sumber is not limited especially as carboxylic acid which is 2-20. For example, acethe ecid. propionic acid, butaneic acid, isobutyric acid, 2-ethyfbutanoic acid, Octanoic acid, 2-

preferred that it is 0.01 to 3 weight section to the polyviryi-acetal resin 100 above-mentioned weight section. When a preventive effect of a white blush mark according that they are less than 0.01 weight 10075]As for an addition of carboxylic acid whose above-mentioned carbon numbers are 2-20, it is compatibility with resin becomes poor and transparancy has a possibility of a problem arising or sections to moisture absorption may become insufficient and exceeds three weight sections. promoting degradation of resin. It is 0.05 to 1 weight section more preferably.

hydrophobicity and compatibility with polyvinyl-anetal resin may worsen. Carbon numbers are 6-12 formula (II), when a carbon number of the above-mentioned alignatic series system hydrocarbon group or the above-mentioned aromatic system hydrocarbon group exceeds 18, it may become  $10376 \mathrm{Jm}\ \mathrm{R}^3$  and  $\mathrm{R}^4$  of phospharic acid which are expressed with the above-mentioned general

expressed, it is not limited, but can use phosphoric acid generally used, and specifically, For example, methylphosphoric scid, ethylphosphoric scid, prapylphosphoric scid, isopropylphosphoric scid, Butyl diethylphosphoric soid, diisopropylphosphoric soid, dioctylphosphoric soid, diphenylphosphoric soid, phosphate, lauryphosphate, stearyphosphoric acid, 2-ethylhexyl phosphoric acid, Di(2-ethylhexyl) dibenzylphosphovie soid, etc. are merrioned. These may be used alone and may use two or more [0077]Especially as phosphoric acid which is the above-mentioned general formula (II) and is phosphoric acid, isodecyf phosphoric acid, phenylphosphoric acid, dimethylphnsphoric acid,

[0378]As for an addition of phosphoriv and expressed with the above-mentioned general formula (II), above-mantioned weight section. If a preventive effect of a white blush mark by moisture absorption may become insufficient when it is less than 0.01 weight sections, and the amount part of duplexs is exceeded, degradation of resin may be promoted or this phosphoric acid itself may cause a white it is preferred that they are 0.01 - the emount part of duplexs to the polyvinyt-acetal rasin 100 blush mark. It is 0,03 to 1 weight section more preferably.

general formula (III) can be conveniontly used for amine which is compatible in the above—mentioned together with amine which is compatible in resin and a plasticizer. What is expressed with following [8079]Organic acid which is compatible in the above-mentioned resin and a plasticizer is used esin and a piasticizar

Formula 33



61](R<sup>5</sup>, R<sup>6</sup>, and R<sup>7</sup> are a hydrogen atom, an alighatic series system hydrocarbon group whose bon numbers are 1-20 bon numbers are 1-20, or an aromatic system hydrocarbon group whose carbon numbers are 1-20 ang a fermula, respectively.) R<sup>5</sup>, R<sup>6</sup>, and R<sup>7</sup> may be the same, and may differ from each other. 82]When a carbon number of the abover-mentioned sightstic series system hydrocarbon group or abover-mentioned aromatic system hydrocarbon group exceeds 20, it may become hydrochobicity of compatibility with polyvinyi-ecetal resin may worsen. As for any one of R<sup>5</sup>, R<sup>6</sup>, and R<sup>7</sup>, it is for a fong chair, and a hydrocarbon group and R<sup>7</sup>, R<sup>5</sup> and whose R<sup>6</sup> are a hydrogen m and the carbon numbers 1-2 are a hydrocarbon group of the carbon numbers 6-16 more feraby.

ylamine, propyłamine, hexyłamine, octylamine. Decył amine, dodecył amine, tetradecylamine, aniline. 84]As for an addition of amine expressed with the above-mentioned general formula (III), when a sticizer, it is preferred that they are 0.01 - the amount part of duplexe to polyvinyl-acetal resin ben number uses sufferiff sold which is 2-21 as erganic sold which is compatible in resin and a tioned by nitrogen-conteining heterocyclic compounds, such as aromatic amines, such as 1, 2 ne, such as dipropyl amine, dibexyl amine, diootyl amine, and N-methylaniline; Trimethylamme. akuding. The 1st class amins, such as naphthylamine, Dimethylamine, diethylamine, Secondary 83]As amine expressed with the above-mentioned general formula (III), For exemple, they are I weight section. When a preventive effect of a white hiush mark by moisture abanypilen may ome insufficient when it is less than 0.01 weight sections, and the amount part of duplaxs is tiary amine, such as triethylamine, M,N-dimethylhexylamine, M,N-dimethyl octylamine, M,Needed, compatibility with resin becomes pace, a problem may arise in transparency or an alkylamine of the 3rd class, and aniline, and pyridine, etc., and specifically, Methylamine, ethyldecyl amine, M.N-dimethyldodecył amine, N.N-dimethylaniline, and pyridine. etc. are ntioned. These may be used alone and may use two or more sorts together. rlayer may color it it. It is 0.02 to 1 weight section more preferably.

86]As for an addition of amine expressed with general formula (III), when a carbon number uses boxylic acid which is 2-20 as organic acid which is enmartible in the above-mentisned resin and a shoizer, it is preferred that it is 0.01 to 3 weight section to polyvivin-acetal resin 100 weight alon. When a preventive effect of a white blash mark by moisture absorption may become afficient if it is less than 0.01 weight acutions and three weight sections are acceeded, nostlikity with resin becomes poor, a problem may arise in transparency or an interisyer may coior is is 0.05 to 1 weight section more preferably.

88] Amine which is compatible in organic acid, resin, and a plasticizer which are eempatible in resin tal ion which constitutes this metal salt, and its counter ion or [ and / that metai saft which these combined by kneading resin at the time of film production is distributed in rosin, and metal salt of daxs to polyvinyl-acetal reain 100 weight section. When a preventive effect of a white blush mark ucture and a molecular weight suitably and to be used according to the degree of acetafization of a plasticizer which are used as the above-mentioned dispersing agent, Respectively it becomes densation of water is controlled and polyvinyl-acetal resin absorbs moisture, a white blush mark ressed with the above-mentioned general formula (II) as organic acid which is compatible in the burt part of duplexs is exceeded, compatibility with resin becomes poor, a problem may arise in moisture absorption may become insufficient when it is less than 0.01 weight sections, and the face of metal salt of particle state which exists in polyvinyl-acetal resin, and combines with a we-mentioned resin and a plasticizer, it is preforred that they are 0.01 — the amount part of such as suffered but, a carboxyl ion, phospheric acid ion, and AMMONIUM ion, it acts on the 87] As for the above-mentioned organic asid or amine, it is preferred to choose malecular tide state becomes small as the result.] — or it disappears. For this reason, even if local isparency or an interlayer may color it it it is 0.05 to 1 weight section more preferably. above-mentioned polyvinyl-acetal resin or a kind of the above-mentioned plasticizer.

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of an interlayer for glass laminates obtained oen be controlled. Among organic acid which is competible in resin and a plasticizer which are used as the above-mentiuned dispersing agent, especially in phosphoric acid, since e role of a coupling agent of resin and glass is also played, and it has the charecter in which a film and glass do not exfoliate easily even if it absorbs moisture, exfoliation by moisture absorption of a glass laminate periphery can be controlled.

[0089]As for an interlayer for glass laminates, in this invention, it is preferred to contain at least one sort chosen from a group which, consists of alkali metal salt and alkaline earth metal salt as an adhesive strength regulator.

[0090]It is not limited especially as the above-mantinned alkali metal saft and alkaline earth metal saft for axample, safts, zuch as potassium, sodium, and magnesium, are mentioned. As the abovementioned saft, inorganie acid, such as organic seid, chlorides, such as carboxylic acid, such as octylic acid, hexyband, hutanoic acid, acetia acid, and formic seid, and nitrio acid, is mentioned. [0091]As the above-mentioned alkali metal saft and alkaline earth metal saft, it is more preferred that they are akkali metal sait of organic acid of the carbon numbers 5-16 and the alkaline earth metal saft of organic acid of the carbon numbers 6-16, it is magnesium saft of carboxylic acid of the carbon numbers 6-16, or dicarboxylic acid of the carbon.

10092]It is not imited especially as magnesium sait of the above-mentioned carboxytic acid or dicarboxytic acid, Fer example, 2-ethylbutanoic acid magnesium, valeric-acid magnesium, hexanoic acid magnesium, magnesium nonamoute, decanoic acid magnesium, magnesium nonamoute, decanoic acid magnesium, mugnesium nonamoute, decanoic acid magnesium, mugnesium nonamoute, decanoic acid magnesium, etc. are mentioned.

10093]Magnesium, glutaric acid magnesium, adipic acid magnesium, etc. are mentioned.

10093]Magnesium salk of carboxytic acid of the above-mentioned carbon numbers 6-16, or decarboxytic acid. By existing in a form of a salt, without ionizing in a film, and drewing a water molecule near. It is thought that it is possible to stop adhesive strength between an interlayer and glass. It is dependent obtained by this can be made good. Since it is distributed over a membrane surface at high concentration, without condensing in a film and an axcessive white blush mark at the time of moisture abscration is not caused white a small quantity shows an adhesive strength adjustment effect, it is desirable.

[0094]As for the above-mentioned alkali metal salt and alkaline carth matal salt, it is preferred that particle diameter is 3 micrometers or less, and they are 1 micrometer or less more preferably. Since it will grow up to be even a size by which a water molecule which gathered around alkali metal salt and/or alkeline earth metal salt is visualized if it exceeds 3 micrometers, a white blush mark may become remarkable and may not be preferred.

\$1095]k is not limited especially as a means for the above-mentioned particle diamater to be 3 micrometers or less. For example, even if it is a method of using a compound which is easy to dissolve in polyvinyi-acetal resin or a plasticizer as an adhesive strength regulator, and a compound which is hard to dissolve in polyvinyl-ecetal resin or a plasticizer, A method of using what it is hard to condense in polyvinyl-acetal resin or a plasticizer, a method of using together a dispersing egent which distributes then, a compatibilizer, etc., etc., are mentioned.

10096]As a compound which is easy to dissolve in the above-mentioned combination, polyvinyl butyral resin is used as polyvinyl-acetal resin, for example, In combination using triethylene glycol 2-cthyl butyrate as a plasticizer, organic acid seit, such as octanoic acid magnesium, neo decanoic acid magnesium, and adixic acid magnesium, etc. are mentioned, and these one sort or two sorts or more are used suitably.

[0081]As a potassium compound which is easy to dissolve in the above-mentioned combination, patassium sait of organic acid, such as cetanoic acid potassium, neo decencic acid potassium, and potassium stearate, cto, are mentioned, and these one sort or twe sorts or more are used suitably, [0098]As a sodium compound which is easy to dissolve in the above-mentioned combination, sodium eath of organic acid, such as sodium extended, neo decane sodium, and sodium stearate, etc. are mentioned, and those one sort or two sorts or more are used suitably.

[0099]As a compound which is during combination of what it is hard to dissolve in the chovermentioned combination, and is hard to condense, magnesium salt of inorganic acid, such as a magnesium chloride end a magnesium nitrate, etc. are mentioned, and these one sort, or two sorts or more are used suitably, for example.

[0100]As a disparaing egent which may be digtributed according to concomitant use, or a compartibilizer, a compound which is hard to dissolve in the above-mentioned continuation. Although

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limited in particular, long chain system organic acid, such as alcohols and octanoic acid, such as ano! and cetyl alcohol, and nonanono acid, is mentioned, and these one sort or two sorts or more used suitably, for example.

spound which it ranks second and is hard to condense in polyvinyl-acetal resin and a plasticizer is theirer also in various above-mentioned methods is the most preferred, and a method of using a 01]A method of using a compaind which itself tends to dissolve in polyvinyl-acetal resin or a

acture of a diester system compound. Since it can distribute stably and uniformly and can exist in 02]As the above-mentioned alkeli metal salt and alkaline earth metal salt, when using a diester in by having a structure similar to an acid component of a diester system compound used as a tem compound as a plasticizer, it is preferred that it is what has an acid component and the sticizer, aging is not caused.

of the carbon number 5 or carboxylic acid of 8 contain as an adhesive strength regulator. Since a ig hereafter called "3GH") or a dihexyl herse mackerel peat (henceforth "DHA"), by making metal nosforth "3QO") as a plasticizer for same reason, it is preferred that metal zalk of carboxylio acid 03]As the above-mentioned plasticizer, triethylene glycol di-2-ethy: butyrate, When using (it also vention from a white blush mark and prevention from a fall with the passage of time of adhesive of adhesive strength of an interlayer and glass with the passage of time can be prevented and angth can be reconciled, it is desirable. When using trictiviene giveol di-2-ethylhaxanoete he earbon numbers 6-8 contains. When using tetrsethylene Gloo Lod'z 2-othylbexanoate nceforth "4GO") as a plasticizer, it is preferred that metal salt of the carbon number 6 or boxylic acid of 7 contains.

as di-haptanoate (3G7) or tetraethylene glycol di-haptanoate (4G7). It is preferred to use a side 04]In order to prevent hydrolysis by heat at the time of film production of polyvinyl-acetal resin sticized [ above-montioned ] as much as possible, it compares with a plasticizer like tristly/ene in type piesticizer fike 3GH which cennot cause hydrolysis easily, 3GO, and 4GO, or a horse skerel peat system type plasticizer like DHA.

arttage of being hard to cause hydrolysis, as compared with 307 and 467 grade which an organic npared with 3GH, since the above-mentioned 3GO or 4GO are high boiling points, they have the we-mentioned 3GH, 3GO, 4GO, and OHA. Since the characteristic which above-mentioned 3GH, 06]Above-mentioned 3GH, 3GO, 4GO, and DHA may use together with other plasticizers which 4GO, and DHA have can weaken with other plasticizers if it is 50 % of the weight or more, an à ingredient of a raw material is a side chain type, and are straight chain types. For example as 05]Above-mentioned 3GH has a track record lang as a plasticizar for interlayers, and has the ntioned 3GH, 3GO, 4GO and DHA, and other plasticizers is not limited, it is preferred that the ount of concomitant use of other plasticiners is less than 30% of the weight of a plasticizer of y be used alone and mentioned later. Although a concomitant use rate in particular of above set of an adhasive strength regulator used corresponding to these may not no longar be antage of being hard to vaporize at the time of film production or doubling processing.

f metal sak (carbon number 5), haxanoic acid (2-ethythutanoic acid) metal sak (carbon number 6), we-mentioned plasticizer. The above-mentioned carboxylic acid may be a straight chain type, and 07]Metal salt of carboxylic ecid as an adhesive strength reguiator used when setting a plasticizer he above-mentioned interlayer as a specific thing. Although not limited in partiouser, pentanoio stanois aeld metal salt (oarbon number 7), optanois aeld metal salt (carbon number 8), etc. are ntioned, and those one sort or two sorts or more are suitably used according to a kind of the y be a side chain type.

08]The maisture resistance of an interlayer obtained may become insufficient in case of metal salt passage of time may become insufficient conversely in case of metal salt whose carbon number y become large, and a fall preventive effect of adhesive strength of an interlayer and glass with 1se carbon number of the above-mentioned carboxylic acid is too small, generating of elthism parboxylic acid is too large.

09]Metal sait of carboxylic acid as the above-mentioned adhesive strength regulator, Although it r be used independently, respectively, formic acid magnesium, magnesium acetate, A metal salt, tem adheaive strongth regulator of carboxylic acid of the carbon numbers 1-4 like magnesium

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adiesive strength regulator, the addition has 0.01 to 0.2 preferred weight section to polyviny-apetal resin 100 weight section. Since an adhesive strangth adjustment effect will be last if it is less than regulators, such as a denaturation silicone oil system adhesiya strength regulator mantionad later. carrying out bleed out, there is a possibility that adhesive strength of an interlayer and glass may 0.01 weight sections, penetration—proof performance of a glass laminate obtained may fall. If 0.2 OTO When adding the above-mentioned alkali metal salt and/or alkaline earth metal salt as an weight sections are exceeded, while spoiling the transparency of a glass faminate produced by proparoate and magnesium futanoste; it may be used together with other authesive strongth decline too much. It is 0.03 to 0.03 weight section more preferably.

earth metal salt as a neutralizer of an acid catalyst of chloride etc., and a reaction of polyvinyl-acetal polyvinyi-acetal resin, The above-mentioned metal sait may have contained in various raw materials. cothers / in a case of adding as the above-mentioned adhesive strength regulator } in a reaction of water, etc. which were used in a case where it originates in having used alkali metal salt and alkaline potassium element concentration, since a white blush mark happens casily also when the above $\sim$ [0111] As for sodium concentration, since especially a white blush mark happens easily when the above-mentioned alkali metal salt is sodium sait, being set to 50 ppm or less is preferred. As for 10112]The above-mentioned alkali metal salt and/or alkaline earth metal salt. Sulfuric acid used resin. Alkali metal saft and alkaline earth metal salt as the above-mentioned neutralizer can be mentioned alkali metal salt is notassium salt, being set to 100 ppm or less is preferred, diverted to some other purpose as an adhesive strength regulator.

[018]An interlayer for glass laminates of this invention consists of a plastic resin film which consists of additive egents, such as an above-mensioned dispersing agent end an adhosive strength regulator. by the above-mentioned polyviny/racetal resin, a plasticizer, and necessity.

[0114]As a plasticizer used in this invention, phosphorio acid system plasticizers, such as organic ester system plasticizers, such as a publicly known plasticizer used for this kind of interlayer, for exampie, monobasic acid ester, and polybasic acid ester, an organic phosphorus acid system, en organic phosphorous acid system, etc. are used.

ester obtained by a reaction with organic acid, such as butanaic acid, isobutyric anid, caproin acid, 2ethylbutsnoic anid, heptanoic acid, n-octylic soid, 2-ethylbexyl acid, pelargonic acid (n-nenylic acid), 10115]In the above-mentioned monobasic acid ester, for example Triethylene glycol, Glycol system and docylacid, is preferred. In addition, ester of tetraethylene glycol, tripropylene glycol, and the above-mentioned organic acid is also used.

sebacic soid, and azelair soid, and straight chain shape of the narbon numbers 4-8 or branched state [U18]As the chove-mentioned polybasic soid ester, ester of organic soid, such as adipic soid, alcohol is preferred, for example.

prepylene glycol di-2-cthyl butyrate, 1,4-butylene GURIKORUJI 2-ethyl butyrate, 1,2-butylene-glycol [015]As the above-mentioned phosphoric acid system plasticizer. tributoxyethyl pirosphate, isodecyf compound which consists of dicarbuxylic ecid and menohydric elcohol, or consists of menccarboxylic [0117]As an example of the above-mentioned organic ester system plasticizer, Triethylene glycol diphonyl phosphate, triisepropyl phosphite, etc. are preferred. It is prefarred to blend a diester system [0118]In addition, ethylene glycol di-2-ethyl butyrate, 1,3-propylone glycol di-2-ethyl butyrate, 1,4di-2-ethyiene butyrate, disthylene-glycol di-2-ethyl butyrate, Diethylene-glycol di-2-ethylhexoate, glycol di-n-actoate, Triethylanc glycol di-n-HEPUTOETO, tetraethylane glycol di-n-HEPUTOETO, other dibutyl sebscate, dioctylazelate, and a dibutyloarbitol horse mackerel peat are used suitably. dipropylene głycoł di-2-ethył butyrata. Triethylene głycoł di-2-ECHIRUPENTOETO, tetraethylene 2-ethyi butyrate, triethylene glycol di-2-ethylhexeate, Triethylene glycol dicaprylate, triethylene glycol di-2-ethyl butyrate, dethylene-glycoi JRAPURIETO, etc. are used as a plasticizer. asid and dihydric alcohol in the above-mentioned plasticizer.

penatration resistance of a glass faminate which will be obtained if it is less than 20 weight sections laminates, such as conversion silicons oil for controlling penetration resistance besides a dispersing may fall, if 70 weight sections are exceeded. a plasticizer will carry out bleeding, optical strain may polywhyt-acetal resin 100 weight section, and it is 40 to 60 weight section more preferably. The [0120]As the above-mentioned amount of plasticiters, 26 to 70 weight section is preferred to (01213) this invention, a publicly known additive agent currently used for interlayers for giass become large or the transparency of a resin layer and an adhesive property may fall

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int and an adhesive strength regulator, an ultraviolet ray absorbent, light stabilizer, an antioxidant, urfaperactive agent, and colorant, as an additive agent can be blended.

dified silicone oit, for example, is indicated by JP,55-29950,B, Ether denaturation silicone oil, exter aturation silicone oil, and assine deneturation silicone oil carboxyl modified silicone oil etc. are 22]An spoxy deneturation silicone oil which is not limited especially as the above-mentioned nationed. These modified silicone oil is fluids produced by making a compound which should saturalize to a polysiloxane reaet generally.

23]In this invention, it is following general formula (IV),

(1.5)

25]Epoxy modified silicons oil, following general formula (V) which are expressed with (I and m ress 30 or less positive integer among a formula)

3 H-1(0--110--120)--0--14H 27]Ether madified silicone all expressed with (x and y to which I and m express 30 or less positive iger express 20 or less positive integer among a formula), and following general formula (VI) 28]

(A 1) CH-COOCH, 29]Ester modified silicane oil expressed with i and m express 30 or less positive integer among sJohnser, in this invention, what is expressed with a structural formula of a random copolymer is mula) is used especially preferably. Akthough each modified silicone oil shown by the above~ ntioned general formula (IV), (V), and (VI) is expressed with a structural formula of a block

30]The above-mentioned modified silicone oil may be used alone, and may use two or more serts ether. As for a molecular weight of the above-mentioned modified silicone oil, 800-5000 are ferred. If localization to the surface falls that it is less than 800 and 5000 is exceeded,

npatibility with resin becomes poor, bleed out will be carried out to a membrane surface, and asive strength with glass will fall to it. It is 1500-4000 more preferably.

will be carried out to a membrane surface, and adhesive strength with glass will fall to it. It is 0.03 tion to the polyvinyl-scetal resin 100 above-mentioned weight section. If a preventive effect of a tomes insufficient and exceeds 0.2 weight section, compatibility with resin becomes poor, bleed 31] An addition of the above-mentioned modified silicone of has 0.01 to 0.2 preferred weight. te blush mark according that they are less than 0.01 weight sections to moisture absorption

32]K is not limited especially as the above-mentioned antioxidant, but as a thing of a phenol weight section more preferably.

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JP,11-343152,A [DETAILED DESCRIPTION]

Chemical Co., ££63, Tetrakis [mathylena-3-(3-5'-di-t-butyd-4'-hydroxyphenyl) propionate] methane system, For example, t-butyflydraxytoluene (BHT) ("Sumi Rider BHT (trade name)" by Sumiteme (IRUGA NOx 1010, Ciba-Geigy make) etc. are mentioned.

19133It is not fruited especially as the chove-mentioned ultraviolet ray absorbent, but as a thing of a G&a-Geigy make), A thing of hindered amine systems. such as thing.LA-57 (made by ADEKAAGASU) benzotriazol system. for example, 2-(2'-frydroxy-5'-methylphenyl) benzotriazol (the tinuvin P.) The of benzatriazel systems, such as 2-(2"-hydroxy-3" and 5"-di-amyl phenyl) henzotriezol (the timerin Geigy make), A 2-(2"-hydroxy-3"-t-hutyh-5"-methylpinenyl)-5-chlorobenzotriazole (the tinuvin 326, Gba-Geigy make, 2-(2'-hydroxy-3' and 5'-di-t-buthybhenyi) benzotriazol (the tinuvin 320, Oisa-323, Ciba-Geigy make), etc. are mentioned.

ADEKA stub LA-57" by the Asahi electrification company etc., (trade name) is mentioned. As the above-mantioned surface-active agent, sodium lauryl suffato, alkylbenzene sulfonic acid, etc. are [0134]As the above-mentioned light stabilizer, a thing of a hindered amine system, for example,

extrusion method, the calandar method, the pressing method, casting method, a tubular film process, [0136]thickness of the whole interlayer for glass laminates of this invention -- as a glass faminate plasticizar and if needed I for the specified quantity. A film is produced to a sheet shaped with an [0135]A manufacturing method of an interlayer for glass laminates of this invention. Although not etc., and a resin layer is fabricated, and let this he an interlayer, after kneading this uniformly. limited in particular, other additive agents are blanded with each above-mentioned resin [ a

the minimum .... when required ponetration resistance and weatherability are taken into consideration. generally the range of 0.3-1.6 mm is preferred like thickness in the usual interlayer for glass

[0137] As a glass plate used for the above-mentioned glass laminate, not only an inorganic clear glass mard but orgenio ciear glass boards, such as a polycerbonate board and a polymethylmethaorylate board, can be used.

giass, sheet glass containing a line, heat ray absorption plate glass, and colored sheet glass, etc. may be mentioned, these may be used independently, and two or more sorts may be used together. An inorganio clear glass board and an organic clear glass board may be laminated. Thickness of glass limited, Various inarganic glass. such as float sheet glass, polished plate glass, figured glass, wire [0138]Especially as a kind of the above-mentioned inorganic clear glass board, it is not what is should just be suitably chosen by use and is not restricted in particular by it.

[0139]In order to manufacture a glass faminate of this invantion, a process of the usual glass laminate consists of a resin layer fabricated by an above-mentioned method, and putting this into a rubber bag is adopted. For example, between transparent glass plates of two sheets, pinch an interlayer which and carrying out decompression sucking at about 70-110 \*\*, carry out preliminary adhesion and it. ranks second, it is manufactured by performing this adhesion by a pressure of about ten to 15

kg/cm² at about 120-150 \*\* using a press, using autoelave.

tempersture of 60-100 \*\* at the same time it makes the above-mentioned interlayer which comes at interlayer / glass plate into a rubber bag, and more specifically for example, in autoclave, -For [ 10 to mentioned above in this manufacturing method, and setting up suitably terms and conditions, such as 30 min.des ] grade heat crimping is carried out by a pressure about temperature of about 60-100 \*\*, least to produce a film polyviny butyral resin plasticized between glass plates of a couple intervene earrying out suction deseration by within the limits about the above, it can adjust so thet it may fall and carries out eaction deseration under depoippression. Put a jayered product of a glass plate / a sticking-by-pressure pressure, sticking-by-pressure time, and a decompression degree when and  $i=10\,{\rm kg/cm^2}$ , carrying out suction deseration under decompression of an about [ 500-[0141]By limiting temperature when carrying out heat crimping to the range of 60-100 \*\* as [0140]In a menufacturing method of a glass laminate, heat crimping may be carried out at f00mmilg ], and it carries out by performing deseration and adhesion simultaneously

Example Athough an example is fung up over below and this invention is explained to it in more detail, this invention is not fanited only to these examples. The "part" in an example means a "weight

within the fitness range of a request of adheave strongth of an interlayer and glass.

http://wwwf.ipdl.inpit.go.jp/ogi-bin/tran\_web\_ogi\_ejie?atw\_u=http%3A%2F%2Fwww4.ipdsi... 2009/08/15

26/40 スーツ

opiete, superfluous water washes, and unreacted n-butylaldchyde is flushod. The sodium hydroxide 43]adding the average degree of polymerization 1709, and the degrees of polyvinys alcohol 275g of issolved. Temperature control of the system of reaction was carried out to 12 \*\*, 35% of chloride alysts [ 201 g of ] and the orbutylaideliyde 148g were added, this temperature was held, and the erfluous water. The vinyl-acetal ingredient (degree of acetalization) of this resin was 55.0-mol %, onification of 98.9 mol to the preparation pure water 2890g of Example 1 (1) resin --- warming -ctant was deposited. Then, hold the system of reaction at 45 \*\* for 3 hours, make a reaction tion which is a general-purpose neutralizer neutralized the chloride catalyst, and also white võered polyvinyi butyral resin was obtained through rinsing and desiccation for 2 frours with the vinyl acetate component was 1.1-mol %.

rying out malt kneading of this with a roll mill, with the press—forming machine, press farming was 44](2) Manufacturs above-mentioned polyvinyl-butyrel-resin 100 weight section of an interlayer, rometer and potassium salt of the particle diameter of the sodium salt in an interlayer was less ging of the secondary ion mage using a time-of-dight type secondary-ion-mass-spectroscopy ried out for 30 minutes, and 150 \*\* of 0.76-mm-thick interlayers were obtained. The result of ame of this decomposition product with ultrapure water, it is the method of quantifying by the F-SIMS) device (product [ made by PHEVANS ] TFS-2000 type). The particle diameter of t ing measured the particle diameter of the sodium salt in an interlayer, and potassium salt by mate analysis heats and decomposes a sample with suffuric acid and nitric acid and sets the n 0.5 micrometer. The bottom was a result and 8 ppm in fixed quantity in IGP luminescence er mixing triethylane-glycol-di-2-ethyl-butyrate 40 weight section as a plasticizer and fully mate analysis about the sodium element content of this interlayer. After ICP luminescence "AES method using an IOP-AES device (the "IOAP-378 type" by a depense jar rel ash

utes at 80 more \*\*. Thus, this adhesion was performed for the glass laminate by which preliminary lesion was carried out for 20 minutes on pressure 12 kg/cm² and conditions with a temperature of rubber bag and oarrying out indirect desulfurization mind with the degree of vacuum of 20torr for minutes, it moved to 90  $\star\star$  oven, deserated, and the vacuum press was carried out, holding for 30 ss faminate, an adhesive examination (pan Meru examination) and the moisture-proof white-blushinate with a transparent float glass (30 cm by 30 cmx2.5 mm in thickness), After putting this in in i \*\* in air type autoclave, and the transparent glass iaminste was obtained. About the obtained 45](3) It inserts from both sides using the manufacture above-mentioned interlayer of a glass k sex test were done by the following methods.

46]A quality assessment (1) adhesive-property (pan Menı) examination glass isminate is neglected the which carried out, grade attachment beforehand, as shown in Table 1. This is for the adhesive adjusted to -18 \*\*\*\*0.8 \*\* temperature, and this is struck with the hammer whose weight of a ges of exposure of the film after glass carries out partial avulation is judged with the boundary id is 0.45 kg, and it grinds until the particle diameter of glass is set to 6 mm or icss. And the orgth of a glass plate and an interlayer to judge whether it is in the predetermined range.

5 te 13

バンメラ数 40 çe; \*\* u ೲ 3 温級の総出級 5 C ~ == Ç; (6) (0) © %? 5. ~ О |N

2.55 Ś

0148/Q) The moisture-proof white-blush-mark sox-test rosin layer was cut into the size of 4x4 cm. after was measured using the integral equation turbidity meter (made by Tokyo Denshoku Co., Ltd.). it was immersed in the fon exchange water of ordinary temperature (23 \*\*), and Hayes of 24 hours The result was shown in Table 2.

for the sodium element content in the obtained interlayer, the particle diameter of 3 micromoters and [0149]In preparation of resin of example 2 Example 1, washing and a wazhing process after putting in changed washing and a washing process after putting in a neutralizer in 2.5 hours. In this case, as for changed washing and a washing process after putting in a neutralizer in 3.5 hours. In this case, as for case. The particle diameter of 17 pans and sodium sail, of the sodium element content in the obtained except having changed washing and a washing process after putting in a noutralizer in I four. In this a neutralizer were changed in 1.5 hours, and it carried out like Example 1 except it. In this case, as the sodium element content in the obtained interlayer, the particle diameter of 0.5 micromotor and [0150]In preparation of resin of example 3 Example 1, it carried out like Example 1 except having [0151]In preparation of resin of example 4 Example 1, it carried out like Exemple 1 except having potassum sait of the particle diameter of 13 ppm and sodium seit was less than 0.5 micrometer. the sodium element contant in the abtained interlayer, the particle diameter of less than 0.5 and potassium salt of the particle diameter of 0.9 ppm and sodium salt was less than 0.5 micrometer. 0152]In preparation of resin of comparative example. Example 1, it carried out, like Example. 1 potassium selt of the particle diameter of 3 ppm and sodium salt was less than 0.5 micrometer. interlayer was 8 micrometers.

was 13 micrometers. The evaluation result of the above Examples 1-4 and the comparative examples particle diameter of 35 ppm and sodium salt of the sodium element content in the eletained interlayer except having changed washing and a washing process after putting in a neutralizer in 0.5 hour. The [0153]In preparation of resin of comparative example 2 Example 1, it earlied out like Example 1 and 2 was shown in Table 2.

0.154

[Table 2]

			5	<b>克洛光</b>		-17	五次美
			97	10	*01*	p.,	100
	アナラール医院多数	~	~-	**	ų.	53	*9
ď	子セタール公園(のほ)	85, è	68.6	65.0	ଓ ୬	68.3	81,0
<del>}</del>	平均独合家	1739	1393	1700	1736	3700	3.300
8	どこんでセチート成分 (回立1米)	·-i		7.	:	Ξ	. <u>.</u>
Œ	电影器器(连整器)	2	ŝ	ng.		63	3
2	ナトリウム滋賀(pp8)	œ	Ξ	œ	8, S	1,	<u> </u>
\$	+ 8 2 2 4 4 5 (4 2)		m	8,5	9.9	10	Œ
	カリラム磁性(ppg)	8.5	រព ឃ	33	3, 5	ï	ţ
	カララム監羅 (28)	6,67	() ()	â	3.57	1	į
40	合わなガラスの複数性 Cパンメル版)	œ	se:	æ	တ	301	æ
*	木段准2-4時間後のヘイズ (%)	8	83	Z.	<b>(f)</b>	ij	32
\$	依古料定	0	೦	٥	٥	×	У
				-	1	and and and	and Control of the Co

10155]The maisture resistance excellent in Examples 1-4 is shown so that clearly from the abovementioned result. [0156]adding the average degree of polymerization 1730, and the degree's of polyviny! slophol 275g of it dissalved. Temperature control of the system of reaction was carried out to 12 \*\*, 35% of chloride ostalysts [ 201 g of ] and the n-butylaidshyde 148g were added, this temperature was held, and the saponification of 98.9 mol to the preparation pure water 2890g of Example 5 (1) resin — warming reactant was deposited. Then, hold the system of reaction at 45 \*\* for 3 hours, make a reaction

gree of acetalization) of this resin was 65.0-mo! %, and the vinyl acetate component was 1.1-mol ylaldshyde is flushed, The potassium fydroxide solution which is a general-puspose neutralizer plete, the water of an excess (it is 30 times the amount to resin) washes, and unroacted ntralized the obloride catalyst, and elso white powdered polyvinyl butyral resin was obtained rugh rinsing and desicoation for 2 hours with superfluous water. The vinyi-acetal ingredient

ried out for 30 minutes, and 150 \*\* of 0.76-mm-thick interlayers were obtained. The bottom was a ying out melt kneading of this with a roll mill, with the press-forming machine, press forming was 57](2) Manufacturc above-mentioned polyvinyl-butyral-resin 100 weight section of an interlayer, nental content of this interlayer. The particle dismeter of less than [0.5 micrometer] mum and sr mixing triethylene-glycol-di-2-ethyl-butyrata 40 weight section as a plasticizer and fully oft and 23 ppm in fixed quantity in ICP furninescence ultimate analysis about the potassium assium salt of the particle diameter of the sodium salt in an interlayer was less than 3

utas at 80 more \*\*. Thus, this adhesion was performed for the glass laminate by which preliminary esion was carried out for 20 minutes on pressure 12 kg/cm² and conditions with a temperature of rubber bag and carrying out indirect desulfurization mind with the degree of vacuum of 20torr for minutes, it moved to 90 \*\* oven, deasrated, and the vacuum press was carried out, halding far 30 inste with a transparent float glass (30 cm by 30 cmx2.5 mm in thickness), After putting this in in 58%) It inserts from both sides using the manufacture above-mentioned interlayer of a glass \*\* in sir type autoclave, and the transparent glass laminate was obtained.

petassium elemental content in the obtained interlayer, the partiale diameter of 1 micrometer and fum salt of the partiale diameter of 5 ppm end potassium salt was loss than 0.5 micrometer. 80% preparetion of resh of example 7 Example 5, it carried out like Example 5 except having nged washing and a washing propess after putting in a neutralizer in 3.5 hours. In this case, as for nged washing and a washing process after putting in a neutralizor in 2.5 hours. In this case, as for rometer and sedium salt of the particle diameter of 0.7 ppm and potassium saft was less than 0.5 193n preparation of resin of example 0 Example 5, it carried out like Example 5 except having potassium elemental content in the obtained interlayer, the particle diameter of less than 0.5

opt having changed washing and a washing process after putting in a neutralizer in 1 hour, in this e, the particle diameter of 104 ppm and potassium salt of the potassium elemental content in the 813n preparation of resin of comparative example 3 Example 5. It carried out like Example 5 ained interlayer was 6 micrometers.

apt, having changed washing and a washing process after putting in a neutralizer in 0.5 hour, in this the particle diameter of 220 ppm and potassium salt of the potassium elemental content in the 823in preparation of resin of comparative example 4 Example 5, it carried out like Example 5 ained interlayer was 9 miorometers.

63]About the glass laminate obtained by the above Examples 5-8 and comparative examples 3 and n adhesive examination (pan Meru examination) and a hunidity test are done by the same method the above-mentioned, and the evaluation result is summarized in Table 3, and is shown.

64] 5/e 3]

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## JP,11-443152,A [DETAILED DESCRIPTION]

	はく てくてく てく くてく で 一 で 中央 高音 高音 高音 中央 中央 高音 高音 高音 中央 中央 高さ 高音 でんしゅく あくて えんしゅう かんかん かんかん かんかん かんかん かんかん かんかん かんかん かん	Da.	发烧纸		55 数 数	28 28
		rs	s	3~	23	***
	アセラール基次条数	~9	4	"	ŗ	*
	アセタール代度(88)な)	ନ ନ୍ଦିଆ ଆଧାର	₩S. &	85.8	65.8	6.3
쯗	平均奎合度	2323	1786	90£3	1735	1786
£6.	ビニルアセテート成分 (mo 1%)		<del>-</del> .		Ĭ.,	
×	可图察器 (的强化)	#	ş	#	Ģ	39
*	カリウム液度(ppm)	23	US.	6.7	7 <u>0</u>	228
	カリウム物経 (10名)	×	٦.	<b>ે</b> હ &	ıq	æ
	ナトじウム機関(pys)	3, 7	3,6	9 16	;	;
	ナトリウム税後 (μの)	0.85	<b>€</b> , 5>	8,53	1	i
40	合わせガラスの接着性 (パンノル値)	°	æ	œ	œ	£-a
**	水没後 2 4 時間後のヘイズ (%)	<u> </u>	02	<b></b>	8	£6
*	落分割完	0	٥	ं	×	×

[0165] The moisture resistance excellent in Examples 5–7 is shown so that clearly from the above—

complete, superflusus water washes, and unreacted n-butylaldehyde is flushed. The sodium hydroxida (0166]adding the average degree of polymerization 1700, and the dagree% of polyvinyl alcohol 2.75g of powdered polywinył butyrai resin was obtained through rinsing and desiccation with superfluous water. it dissalved. Temperature copirol of the system of reaction was carried out to 12 \*\*, 35% of chlorida catalysts [ 201 g of ] and the n-butylaldshyde 148g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 \*\* for 3 hours, make a reaction solution which is a ganeral-purpose neutralizer neutralized the ahloride actalyst, and also white saponification of 98.9 mol to the preparation pure water 2890g of Example 8 (1) resin The degree of acetalization of this resin was 65,0-mol %,

forming was cerried out with the press-forming machine, and the interlayer was obtained. The bottom above-mentioned pelyvinyi-butyrai-rosin 100 weight section of the interlayer as a plasticizer, press was a result and 13 ppm in fixed quantity in the IOP luminescence elemental analysis davice about [0167](2) Triethylene-glycoi-di-2-ethyl-butyrate 40 weight section was mixed with manufacture the sodum element content of this interlayer. The particle diameter of the sodium satt in an interlayer was 3 miorometers,

[0169](3), pinching the manusacture above—mentioned interlayer of a glass laminate between the 2.5parformed for 20 minutes on condition of the temperature of 60 \*\*, and pressure " of 5kg/cm, and deaeration under decompression of -600mmHg in autoclave. Heat crimping was simultaneously run-thick float glesses of two sheets, putting this in in a rubber bag, and carrying out suction the giass laminate was produced.

like Example 8 except the temperature at the time of heat crimping having been 80 \*\*. The particle [8169]In production of the glass laminate of example 9 Example 8, the glass laminate was produced diameter of the sodium sait in an interlayer was 3 micrometers.

like Example 3 except the temperature at the time of heat crimping having been 100 \*\*. The particle [0170] in production of the glass laminate of example 10 Example 8, the glass laminate was produced diameter of the sodium salt in an interlayor was 3 micrometers.

[8171]In preparation of reals of comparative example 5 Example 8, except having changed washing was preduced. In this case, the particle diameter of 30 ppm and sodium sait of the sodium element and a washing process for a short time a little, it carried out like Example 8 and the glass laminate contest in the obtained interlayer was 11 micrometers.

72]About the giass laminate obtained by the above Examples 8-10 and comparative example 5, adhesive examination (pan Meru examination) and the heet rosistance tost were done by the wing methods. About the humidity test, it carried out like Example 1.

inate is uniformly hit by a hammer head, the area of the glass piece adhering to the interlayer of a 13]After cooling a valuation method (1) adhesive-property (pan Meru) examination glass laminate tom is carried out with the curvature which is 50 mm in radius, the used automatic hammer ring is laminate is chaerved visually, and adhesive strength (pan Meru value) is judged as confrested 20 \*\* for 2 hours. An automatic hammer ring device is equipped, the whole surface of a glass ice has a hammer head with a blow effective diameter of 5 mm and a weight of 240g, and the dimum of a grade sample is a terr-point method of ten points. Curved-surface shaping of the I the grade sample shown in Table 1. The adhesive strength minimum is one point and the king power of a hammer head oan be adjusted with a spring screw.

nate." The result of the above Examples 8-10 and the comparative example 5 was summerized in 74](2) Take out, end observe the existence of foaming visually, after neglecting the obtained glass hate under 130 \*\* atmosphere for 2 hours based on the heat resistance test. JIS R3205 "glass de 4, and was shown.

	W.	案施例		光紫紫紫
	80	*	e:s (	w.
トリウム微液(ppm)	23	27	2	8
トリウム勉強(418)	m	20	e-0	=
5年ガルスの研究院 (スソメル会)		rt3	ಮಧಿ	ući
3数24%短数のヘイズ (%)	S	<b>%</b>	22	889
5世ガラスの耐熱性試験 5発泡の有線	 #8	 20	<u> </u>	.i 48
	ା	೦	ာ	×

yviny: butyral resin was obtained for this resin through the desicostion under the atmosphere of 50 16]adding the everage degree of polymerization 1700, and the degrees of polyvinyl alcohol 275g of onification of 98.9 met to the preparation pure water 2890g of Example 33 (1) resin ~~ warming ~~ issolved. Tempereture control of the system of reaction was carried out to 12 \*\*, 35% of chioride ylakiehyde was flushed. The pti value was 5.1 when ph of the system at this time was measured. iried with the centering dehydrator and resin of water content was obtained 50%. White powdered alysts [ 201 g of ] and the n-butylaid-hyde 148g were edded, this temperature was held, and the de to complete, the water of the excess (they are 30 times to resin) weshed, and unreacted nctant was deposited. Then, held the system of reaction at 45 \*\* for 3 hours, the reaction was and -700mmHg. The degree of acetalization of this resin was 65.0-mol %.

sodium element content in this interlayer. The particle diameter of the sodium salt in an interlayer ming was carried out with the press-forming neachine, and the interlayer was obtained. The bottom we-mentioned polywinyl-butyrai-resin 160 weight section of the interlayer as a plasticizer, press s a result and 0.7 ppm in fixed quantity in the 10P luminescence elemental analysis device about 77)(2) Triethylone-glycol-dr-2-ethyl-butyrate 40 weight section was mixed with manufacture less than 0.5 migrometer.

78)(3), pinching the manufacture above-mentioned interlayer of a glass faminate botween the 2.5seration under decompression of ~600mmHg in autoclave. Heat crimping was simuitaneausly r-thick float glasses of two sheets, putting this in in a rubber bag, and carrying out suction

formed for 20 minutes on condition of the temperature of 80 \*\*, and pressure  $^2$  of Skg/cm, and ylass laminate was produced gladding the everage degree of polymerizetion 1700, and the degrees of polyvinyl alcohol 275g of

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### JP.11-343152A [DETAILED DESCRIPTION]

reactent was deposited. Then, hold the system of reaction at 45 4\* for 3 hours, and the reaction was \*\* of en excess (they are 30 times to resin), and white powdered polyvaryl butyral resin was obtained it dissoked. Temperature control of the system of reaction was carried out to 12 \*\*, 35% of chioride saponification of 98.9 moi to the preparation pure water 2890g of Example 12 (i) resin --- warming -catalysts [ 20] g of ] and the n-butylaldehyde 148g were added, this temperature was held, and the solution which is a general-purpose neutralizer. Then, it rinsed with water with a temperature of 50 butylaldehyde was flushed, and the chleride catelyst was neutralized using the sodium hydroxide made to complete, the water of the excess (they are 30 times to resin) washed, unreacted nthrough desicoation. The degree of acetalization of this resin was 85.0-mol %.

forming was carried out with the press-forming machine, and the interlayer was obtained. The bottom the sodium element centent in this interleyer. The particle diameter of the sodium saft in an interlayer above-mentioned polyvinyl-butyral-resin 100 weight section of the interlayer as a plasticiner, press was a result and 10 ppm in fixed quantity in the ICP luminescence elemental analysis device about [0180](2) Triethylene-glycol-di-2-ethyl-butyrate 40 weight section was mixed with manufacture

 $\{0161\%3\}$  pinching the manufacture above-mentioned interlayer of a glass laminate between the  $2.5^$ performed for 20 minutes on condition of the temperature of 60 \*\*, and pressure 5 kg/am², and the deaeration under decompression of "600mmHg in autoclave, Heat cramping was simultaneously mm-thick float glasses of two sheets, putting this in in a rubber bag, and carrying out suction glass laminate was produced.

used water with a temperature of 60 \*\* by washing after neutralization. The degree of acetalization of this interlayer was 55.0-mo! %. The bottom was a result and 15 ppm in fixed quantity in the IOP [0182]in example 13 Example 12, the interieyer was obtained like Example 12 except [ ail ] having luminescence elemental analysis device about the sodium element content in this interlayer. The particle diameter of the sodium salt in an interlayer was 4 micrometers.

the sodium element content in this interlayer. The particle diameter of the sodium salt in an interlayer [0183]adding the average degree of polymerization 1700, and the degree% of polyvinyl alcohol 2.75g of complete, superfluous water washes, and urreacted n-butylaldeliyde is flushed, Magnesium hydroxide forming was carried but with the press-forming machine, and the interlayor was obtained. The bottom it dissolved. Temperature control of the system of reaction was carried out to 12 \*\*, 35% of chloride catalysts { 201 g of } and the n-butylaidehyde 148g were added, this temperature was held, and the above-mentioned polyvinyi-butyrai-resin 100 weight section of the interlayer as a plasticizer, press was a result and 0.7 ppm in fixed quantity in the IOP luminescence elemental analysis device about sepanification of 98.9 mol to the preparation pure water 2890g of Example 14 (1) resin --- warming reactant was deposited. Then, hold the system of reaction at 45 \*\* for 3 hours, make a reaction 19184](2) Triethylene-glycol-di-2-ethyl-butyrate 40 weight section was mixed with manufacture superfluous water, and white powdered polyvinyl butyral resin was obtained through desiecation. which is a general-purpose neutralizer neutralized the chloride catelyst, and also it rinsed with was less than 0.5 micrometer.

(0.85%), pinching the manufacture above-mentioned interlayer of a glass laminate between the  $2.5^\circ$ porformed for 20 minutes on condition of the temperature of 60 \*\*, and pressure 2 of 5kg/sm, and deseration under decompression of "600mmHg in autoclave. Heat crimping was simultaneously nmr-thick float glasses of two sheets, putting this in in a rubber bag, and oarrying out suction the glass laminate was produced.

[0166]About the glass faminate obtained in above Examples 11-14, the heat resistance test was done by the fallowing methods. About the humidity test, it carried out like Example 1. However, Example 14 cerried out only the humidity test.

heated in a 150 \*\* oil bath for 60 minutes, and the existence of degradation of resin was observed. [0187]The resin 1g was put into the valuation method (1) heat resistance test usual tast tube, it The result of the above Examples 11-14 was summarized in Teble 5, and was shown.

Table 5]

		**	米海沙	
		65 24 54	00 ent	ere out
トリウム機能(psw)	9,7	2	57	į. O
トリウム粒径(412)	8,55	352	٧	G 83
縣性減數	器器ない	照25 287	な器に	į
後後25時間後のヘイズ (光)	65 FT	25	8	*
会物定	0	0	O	1

89] adding the polyving alcohol 275g of the everage degree of polymerization 1700 and 0.1% of the 30 times to resin) washes, and unreacted n-butylaidehyde is flushed, After neutralizing a chinride d the system of reaction at 45 \*\* for 3 hours, make a reaction complete, superflueus water (they ight of the degree 2 and sodium scetate content of saponification of 96.3 mol to the preparation afyst using sodium hydroxide which is a general-purpose neutralizer, superflucus water washed ylaldehyde 148g were added, this temperature was held, and the reactant was deposited. Then, 'e water 2880g of Example 15 (1) resin --- warning --- it dissolved, Temperature control of the tem of reaction was parried but to 12 \*\*, 35% of chibride catalysts [ 201 g of ] and the nwhite powdered polyvinyl butyral resin was obtained through desiccation. The degree of stalization of this resin was 65-mol %.

ming was carried out with the press-forming machine, and the interlayer was obtained. The hottom s a result and 8 ppm in fixed quantity in the ICP luminescence elemental analysis device about the we-mentioned polyvinyl-butyral-ream 100 weight section of the interlayer as a plasticizer, press 90](2) Triethylena-giyeal-di-2-ethyl-butyrate 40 weight section was mixed with manufacture fann efement content in this interlayer,

92]adding the polyvinyl sloobol 275g of the average degree of polymerization 1700 and 0.1 % of the \$11The interlayer was obtained like Example 15 except having used the polyvinyl alcohol 275g of example 18 avarage degree of polymerization 1700 and 0.4 % of the weight of the degree % and lium acetate unitent of seponification of 88.9 mol. The degree of ecetalization of this interleyer s 65.0-moi %. The bottom was a result and 13 ppm in fixed quantity in the 10P fuminescence mental analysis device about the sodium element content in this interlayer.

edered polyvinyi butyrsi resin was obtainad through desiocation. The degree of seetalization of this 30 times to resin) washes, and unreacted n-butyfaidenyde is ffushed, After neutrafizing a chlorida f the system of reaction at 45 \*\* for 3 hours, make a reaction complete, superfluous water (they ght of the degree % and sodium asstate centent of saponification of 98.9 mal to the preparation alyst using octanoio acid magnesium which is a neutralizer, superfucies water washed and white ylaldahyde 148g were added, this temperature was held, and the reactant was deposited. Then, e water 2890g of Example 17 (1) resin --- warming --- it dissolved. Temperature control of the tom of reaction was varied out to 12 \*\*, 35% of phlaride catalysts [ 201 g of ] and the rein was 65-mul %.

ning was carried out with the press-forming machine, and the interlayer was obtained. The bottom s a result and 2 ppm in fixed quantity in the ICP luminescence elemental analysis device about the wa-mentioned polywiny-butyral-resin 190 weight section of the interlayer as a plasticizer, press 93](2) Triethylene-glycol-di-Z-ethyl-butyrate 40 weight section was mixed with manufacture lium element content in this interlayer.

at the sodum element content in this interlayer. Using the interlayer obtained in above-mentioned 94]The interlayer was obtained like Example 17 except having used ethyleneoxide as a nautralizer in example 18 chloride catelyst. The degree of acetalization of this interlayer was 85-mol %. The tom was a result and 2 ppm in fixed quentity in the ICP luminescence elemental analysis device unples 15-19, the same beat resistance test as Example 11 and the same humidity test as imple 1 were done, and the result was aknown in Table 6.

Sie 6)

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		34	美施例	
		9 * 5 1	Pro ,d	٠. در
リゼニルアルコームの修設 -トリウム会有数(変数%)	9.3	* 0	્ર છે	o'
トサウム線道(ppm)	est)	Œ	54	\$1
・トリウム粒屋(ル3)	80 G	64	6.9	÷.
KUIK	跳びる	器はい	設はない。	説に
投資 2 4 毎個後のヘイズ   (※)	22	<b>8</b> 9	<b>::</b> :	<u>e</u> .
合物定	ು	0	୍	្
	CARLLY LAND	あるち しんしし からから	The same of the same of	

+ 4 6 \* .82

flushed. The sodium hydroxide solother which is a general-purpose neutralizer neutralized the chloride tomperature was held, and the reactant was doposited. Then, hold the system of reaction at 43 \*\* for catalyst, and white powdered polyvinyl butyral resin was obtained through rinsing and desircation for alcohol 275g of 98.9 mel to the preparation pure water 2890g of example 19(1) polyvinyl-acetal rasin --- warming --- it dissolved. Temperature central of the system of reaction was cerried out to 12 \*\*, 2 hours with still more superflueus water. The amount of 64-mol % and survival acetyl groups of the 10196]adding the average degree of polymerization 1700, and the saponification degrees of polywinyl 35% of the weight of chloride catalysts [ 201 g of ] and the n-butylaldehyde 148g were added, this 3 hours, make a reaction complete, superfluous water washes, and unreacted n-outylaidehyde is degree of formation of everage butyral of this resin was 1-mol %.

laminates 0.08 copy of outsnoic acid magnesium is added as a plasticizer to emount of residual acetyl minutes at 150 \*\* using the press-forming machine, and the interlayor for glass laminates of 0,76 mm [8197](2) the polyvinyl butyral resin (average-degree-of-polymerization 1700 and degree % of 64 mol content in the obtained interlayer for glass laminates was I micrometer. It was 0.9 micrometer when regulator. After fully earrying out melt kneading with a roll mill, press forming was parried out for 30 of average thickness was obtained. The partiele diameter of 10 ppm and sodium salt of the sodium the particle diameter of the magnesium salt which exists in an interlayer was measured using the groups 1 mol %100 copy as 40 copies of triethylene glycol G"2-ethyl butyrate, and an achesive of formation of average butyral,) ebtained by the manufacture above of the interlayer for glass time-of-flight type secondary-jon-mass-spectroscopy device (TOF-SMAS).

[0198]i3) The interlayer for glass laminates obtained by the manufacture above of the glass laminate. the glass laminate by which preparative pressure arrival was carried out fer 20 minutes on condition degree of vacuum of 20torr for 20 minutes, it moved to over, deserated, and the vacuum press was carried out, holding for 30 minutes at 90 more \*\*. Thus, this sticking by pressure was performed for of the temperature of 135 \*\*, and pressure 12 kg/cm² in air type autoclave, and the glass faminate thickness), putting this in the rubber bag and carrying out indirect desulfurization mind with the After having put from the both sides with the transparent float glass (30 em by 30 cmx3 mm in wes obtained.

lanunates and the glass lanurate were obtained like Example 19 instead of U.OS copy of octanoic acid t was 0.5 micrometer when the particle diameter of the magnesium saft which exists in the interlayer magnosium as an adhesive regulator except having added 0.09 copy of neo decenoic acid magnesium. .01993in manufacture of the interlayer for example 20 glass laminates, the interlayer for glass

laminates and the glass laminate were obtained like Example 19 instead of 008 copy of octanoic acid 0200]In manufacture of the interlayer for example 21 glass laminates, the interlayer for glass for glass laminates obtained above was measured like Example 19.

micrometers when the particle dameter of the magnesium salt which exists in the interlayer for glass magnesium as an adhesive regulater except having added 0.04 copy of magnesium chloride. It was 2 9201]In manufacture of example 22 polyvinyl-acetal resin, as a neutralizer, Instead of sodium faminates obtained above was measured like Example 19.

ydroxide solution, the everage degree of polymerization 1700, and's of the powdered amount white

JP,11-343152,A [DETAILED DESORIPTION]

yoiry! butyral resin of degree % and survival acety! groups of formation of average butyral of 64 for 1 mol were obtained fike Example 19 except having used besic magnesium carhonate. ssequently, instead of 100 copies of polyviny! butyral resin obtained in Example 19 in manufacture the intertayer for glass laminates. The intertayer for glass laminate were ained like Example 19 using 100 copies of polyviny! butyral rasin obtained above except not having 100 copies of polyviny! butyral rasin obtained above except not having 10 ppm and excline is used or of ortanoic acid megnesium as an adhesive strength regulator. The particle demoter 10 ppm and sodium said of the sodium context in an interlayer was less than 0.5 micrometer. It is 2 micrometers when the particle diameter of the magnesium sait which exists in an interlayer is measured like Example 19.

02]th manufacture of example 23 polyvinyl-acetal resin, as a neutralizer, Instead of sodium froxide solution, the average degree of polymerization 1700, and% of the powdered amount white ryrinyl butyraf resin of degree % and survival acetyl groups of formation of average butyral of 64 and formation of 65 and formation of 65 and formation for as a formation for formation formation for formation for formation formation for formation formation for formation format

03]In manufacture of the interlayer for comparative example 6 glass laminates, as an adhesive ulator. The interlayer for glass laminates and the glass laminate were obtained like Example 19 lept having added 0.04 copy of magnesium acetate which is hard to dissolve in polyvinyl butyral in or a plasticizer instead of 0.08 cepy of cotanolo acid magnesium. It was 10 micrometers when particle diameter of the magnesium saft which exists in the interlayer for glass laminates obtained we was measured like Example 19.

04]In manufacture of the interlayer for comparative example 7 glass laminates, as an adhasive ulator. The interlayer for glass laminates and the glass laminate were obtained like Example 19 uspt flaving added 0.05 copy of butanoic acid which is 0.04 copy of magnesium acetate and shert in system organic acid which are hard to disactive in polyvinyl butyral resin or a plasticizer instead 3.08 copy of octanoic acid magnesium. It was 4 micrometers when the particle diameter of the gressium salt which exists in the interlayer for glass laminates chtained above was measured like mpie 18.

05]The humidity test of the interlayer for glass faminates obtained by Examples 19~23 and the aparative exemples 6 and 7 was done like Example 1. The result was as being shown in Table 7, 06]

ole 7]

		**	×8.7			25	288
	¢n ~≺	2.0	ñ	274 673	24 24	w	ĸ.
アセクール勘役条数		~	৵	*	*	~	7
年均ブテラー4位後 (セル第)	94.0	0.50	4	£.0	6.5	95.0	34.0
気停アセテル <b>名名</b> (でんち)	6,7	Q :-	e	e.	8.	9.7	. c
學也被後令後	1703	1780	232.	1200	: 783	2703	3.206
可能效果 (金属部)	¥	÷	÷	*	3	₽	₽
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ップネンウム整体制度 (電解的)	80 K	8	£.		-3	13 15	: : :
マグキンワム戦の被後 (AB)	o, d	υ: 	es Ge	હ જ	80 80	56.0	ر ج
(第24時間後のヘイズ (33)	13,8	8.4	8.8	*	38. 2.5	% %	57.2
**	۵	٥	្	೧	្	×	×

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[0207] The glass laminate of Examples 19-23 by this invention showed the outstanding moisture resistance so that clearly from Table 7. On the other hand, the glass laminate of the comparative examples 6 and 7 with the particle diameter of greater than 3 micrometers of the magnesium saft which exists in a glass laminate interlayer had bad moisture resistance.

Water 2900 weight section, it heated and dissolved in 95 \*\*, having supplied degree\$ of polywing alcohol of seponification of 982 mol 193 weight section, it heated and dissolved in 95 \*\*, having supplied degree\$ of polywing alcohol of seponification of 982 mol 193 weight section (4.5 mol of viny alcohol considerable amount) to the reactor with an aglating device, and agitating it with the avarage degree of polymerization 1700. After cooled this solution at 30 \*\*, heaving added 35-% of the weight chloride 208 weight section 120 and and n-bubladelynde 152 weight section (2.1 mol) lowering solution temperature subsequently to 2 \*\*, holding this temperature and polywiny butyral resh's depositing temperature up of the solution temperature was carried out to 30 \*\*, and it was held for 5 hours. After helding, sodium bicarbonate 158 weight section (1.8 mol) was added it neutralized, rinsing and desiccation were performed, and the degree% of polywiny butyral resin of bublang of 65 mol was obtained it was 50 pum when the sodium enotest of the obtained polywiny butyral rosin was measured by 10P emission spectrometry. The particle dismeter of sodium salt was 12 micrometers.

[1209]#Production of a rosin layer) Polywiny-layer-resin 100 obtained weight section. Tricthylene—glycol-di-2--ethyl-butyrate 40 weight section, ethylenomental social 0.05 weight section.

[0269][Production of a resin layer, Polyvinyl-butyral-resin 100 obtained weight section, Triethylene gycol-di-2—ethyl-butyrate 40 weight section, ethylenediaminetatraecelio acid 0.05 weight section, Press forming of the kneaded material produced by supplying 2-ethylbutanole acid magnesium 0.04 weight section and madified silicons vil 0.05 weight section to a roll mill, and kneading them was carried out for 30 minutes on condition of 150 \*\* and 120 kg/om² with the press-forming machine, and the 0.3-mm-thick resin layer was obtained. About the obtained resin layer, the same humidity test as Example 1 was done. As modified silicone oil, what is expressed with a following ehemical formula was used.

[0210]

[Formula 7]

$$CH_3 - CH_3 - CH_3 - CH_3 - CH_3$$
 $CH_3 - CH_3 - CH_3 - CH_3 - CH_3$ 
 $CH_3 - CH_3 - CH_3 - CH_3 - CH_3 - CH_3$ 

10211]Instead of example 25 ethylenediaminetetrascellur-ecid 0.05 weight section, the resin layer was obtained and eveluated like Example 24 except having used selicylaldehyde 0.08 weight section. The result was shown in Table 8.

[0212]Instead of example 28 othylopediaminetetraneliku-acid 0.05 weight section, the resin layer was obtained and evaluated like Example 24 except having used oxalic acid 1.0 weight section. The result was shown in Table 8,

[0213]Instead of example 21 ethylenediaminetetraeceticr-ecid 0.05 weight section, the resin layer was obtained and evaluated like Example 24 except having used 1,10-phenanthroline 0.03 weight section. The result was shown in Table 8.

10214]Instead of example 28 ethylenediaminetotrescetic—sold 0.05 weight section, the resin layer was obtained and evaluated like Example 24 except having used acetylacetone 0.3 weight section. The result was shown in Table 8.

10215]The resin layer was obtained and evaluated like Example 24 except not having used

comparative example & ethylenediaminetatracetic-acid 0.05 weight section. The result was shown in Tabia 8.

[0216]Instead of comparative example 9 ethylenediaminetetraacetic-acid 0.05 weight section, the resh layer was obtained and evakuated like Example 24 except heying used acetone 0.1 weight section. The result was shown in Table 8.

(10217) (Table 8] http://www.hpdl.inpit.go.jp/egi-bin/tran\_web\_cgi\_ejje?atw\_u=http%3A%2F%2Fwww4.ipdli... 2009/06/15

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<b>大阪教2.4 郡</b> 院		28, 9	28.3	35. 8	29 - 52 20 - 52	3 B. B	27.8	# 55 # 55
が次くない。	が発験)	4.65	9, 0,5	9. eg	. G.	8, 65	0, 0 0, 0	0. 0
245.00 250.00 25	(多数器)	B. C.	0. 24	9, 6, 4	9,04	6,04	1	3
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名体形成化合物	数数	4407717四路陸續	<i>ትየ\$፱፻፴፻</i> ፻፻	シェク数	£, 10-74378952	アセテルアセトン 0.3	24 7-	7.44.6
ジエステル	#   (2)   (3)   (3)   (3)   (4)   (4	46	3	۵.	48	4.6	48	4.0
ブチラール		196	1.00	100	1 O E	1 8 4	190	139
		* %	23	2% 20	23 7-	æ ∾	790	Ø

orification of 98.9 mcf to the preparation pure water 2890g of example 29(1) polyvinyl-acetal resin 18]adding the average degree of polymerization 1700, and the degree's of polyvinyl elechol 275g of icoation for 2 hours with superfluous water. The amount of 64-mol % and survival scetyl groups of ction at 45 \*\* for 3 hours, make a reaction complete, wash, and Then, unreacted n-butylaidehyde --- it dissolved. Temperature control of the system of reaction was carried out to 12 \*\*, perature was held, and the reactant was deposited. With superfluous water, held the system of oride catalyst, and also white powdered polyvinyi butyral resin was obtained through rinsing and shout. The sodium hydraxide solution which is a general-purpose neutralizer neutralized the g of 35-% of the weight chloride catalysts and the n-butylaidehyde 148g were added, this degrae of formation of average butyral of this resin was 1-mol %.

ount of residual acetyl groups 1 mal %100 aapy, as a plasticizer 40 copies of triathylene glysol di-19](2) manufacture profit \*\*\* polyvinyl butyral resin (average-degree-of-polymerization 1700 degree % of 64 mal of farmation of average factoral.) of the interlayer for glass laminates To ithyl butyrate, And 0.13 copy of dimethyl octylamine is added as 0.75 copy of

lecylbenzenesuffenic acid, and amine as organic acid, After carrying out melt kneading enough with utes at 80 more \*\*. Thus, this adhesion was performed for the glass laminato by which preliminary minutes, it mayed to 90 \*\* oven, deaerated, and the vacuum press was carried out, holding for 30 cm2 in air type autoclave, and the glass laminate was obtained. About the obtained glass laminate rubber bay and carrying out indirect desuifurization mind with the degree of vacuum of 20torr for ission was carried out for 20 minutes on condition of the temperature of 135 \*\*, and pressure 12 an the sodium content of the obtained interlayer was measured by IOP emission spactrometry. It s measured using the time-of-flight type secondary-ton-mass-spectroscopy device (TOF-SIMS). off mill, press forming was cerried out for 30 minutes at 150 \*\* using the press-forming machine, the interlayer for glass laminates of 0.76 mm of average thickness was obtained. It was 50 ppm inate with a transparent float glass (30 cm by 30 cmx3 mm in thickness). After putting this in in s 4 micrometers when the particle diameter of the sodium element which exists in an interlayer 20](3) It inserts from both sides using the manufacture above-mentioned interlayer of a glass

ted instead of 0.75 copy of dodecylhenzenesulfonic soid as organic acid, Except having added 0.35 aired like Example 29 and the evaluated result was shown in Table 9. It was 5 micrometers when 21]In manufacture of the interizyer for example 30 glass laminates, 0.30 copy of octanuis acid is ly of decyl amina listead of 0.13 copy of dimethyl octylamine as amine, the resin layer was priayer, the humidity test was done like Example 1 and the result was shown in Table 9.

particle diameter of the sodium element which exists in the obtained interlayer was measured like 22]In manufacture of the interlayer for example 31 glass laminates, except having added 0.20 mapy IK2-ethylnexyl) phosphoric soid instead of 0.75 copy of dodecytherzenesulfonic soid as organic unple 29.

d, the resin layer was obtained like Example 29 and the evaluated result was shown in Table 9. It s 2 micrometers when the particle diameter of the sodium element which exists in the obtained ırlayer was measured like Example 29. 23]In manufacture of the interlayer for comparative example 10 glass laminates, except not having and the eveluated result was shown in Table 9. It was 20 micrometers when the particle dameter ted the organic ecid and amine of the dispersing agent, the resin layer was obtained like Example

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JP,11-343152,A [DETAILED DESCRIPTION]

10224fm manufacture of the interlayer for comparative example 11 glass laminates. except not having hours in 3 hours, the resin layer was obtained like Examplo 29 and the evaluated result was shown in measured like Example 29. It was 13 micrometers when the particle diameter of the sodium element added the organic acid and amine of the dispersing agent, and heving changed washing time from 2 of the sedium element wixes exists in the obtained interlayer was measured like Example 29. Table 9. It was 30 ppm when the sodium centent which exists in the obtained interlayer was wes measured like Example 29.

Table 93 0225

		रमर	案機器		32.	光数弦
		870 0-4	\$2 60	~~ en	æ 	~~ •
-	アセクール基技系数	*	34	***	~*	-
	学均ブチラーの化波 (モルS)	64, 9	64,8	6,18	64.0	84.0
築	数学アセチル類型 (モル光)	6.3	0.7	°.	6.7	8
3	學均差合度	3706	3325	1730	1730	:780
<b>E</b>	可要析器(重然器)	9	<del>44</del>	\$	Ĉ,	<b>\$</b>
455	ナトリウム後漢(apu)	Ģ	56	38	8	۶
	ナトリウム設備 (28)	~	(63	ক্ষ	ž	Ē
à.	水浸油とす路線後のヘイズ	88	83	20	35	62°
1	緣合程定	ं	ာ	٥	×	×
l	The state of the s				The same of the same	· · · · · · · · · · · · · · · · · · ·

700, After cooled this solution at 30 \*\*, having added 35-% of the weight chloride 196 weight section alcohol of sanonification of 99.2 mal 198 weight section (4.5 mal of vinyl alcahol considerable amaunt) were performed, and the degrees of polyviny! butyral resin of butyrah-izing of 85 mol was obtained. It 10227](Production of a resin fayer) Polyvinyl-butyral-resin 108 obtained weight section, Triethylenegycol-dr-2-ethy-butyrate 40 weight section. p-toluonesulfanic-acid 0.43 weight section. And press subsequently to 2 \*\*, holding this temperature and polyviny butyral resin's depositing, temperature forming of the kneaded material produced by suppiying hexylamin≥ 0.23 weight section to a roll mill, :0226]Example 32 composition / preparation (composition of polyvinyl butyral rasin) ton-exchangewas 50 ppm when the sodium content of the obtained pulyviny! butyral resin was measured by IOP up of the solution temperature was carried out to 30 \*\*. and it was held for 5 hours. After holding, sodium bicarbonata 147 weight section (1,7 mol) was added, it neutralized, rinsing and desiccation water 2900 weight section, it heated and dissolved in 95 \*\*. having supplied degrees of polyvinyl to the reactor with an agitating device, and agitating it with the average degree of polymerization and kneading it was carried out for 10 minutes on condition of 150 \*\* and 120 kg/cm² with the (1.8 ma) and n-buty/sidelyyde 152 weight saction (2.1 mal), lowering solution temperature emission spectrometry. The particle diameter of sodium salt was 12 misrometers.

evaluated like Example 32 except having used tetradecylamine 0.49 weight section. The result was [0228]Instead of example 33 hexylamine 0.23 weight section, the resin layer was obtained and shown in Table 10. shover in Table 10.

press-forming machine, and the 0.8-mm-thick resin layer was obtained. The obtained resin layer was

used and the moisture-proof whito-blush-mark sex test was done like Example 1. The result was

(0228) Instead of example 34 p-tolusoesuffonc-acid 0.43 weight section, the resin layer was obtained and evaluated like Example 32 except having used dodecythenzenesulfanic acid 0 75 weight section. The result was shown in Table 10.

obsersationic—acid 0.43 weight soction and hexylamine 0.23 weight section except having used 0230]The resin layer was obtained and evaluated like Example 32 instead of example 35 phttp://www4.indl.inpit\_go.jn/cgi~bin/tran.wab\_egi.ejia?etw\_u=http%3A%2F%2Fwww4.ipdl.i.. \_2009/08/15

lecyßenzenesulfanic acid 0.15 weight section and decyl amine 0.07 weight section. The result was

leoyBenzenesulfonia gold 0.75 weight section and decyl emine 0.36 weight section. The result was Janesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section except having used 31jThe resin layer was obtained and evaluated like Example 32 instead of example 38 p-

dacylbanzenesulfonic soid 0.75 weight section and dodsoy! smine 0.42 weight section. The result renesultonic—soid 0.43 weight section and hexylamine 0.23 weight section except having used 32]The resin layer was obtained and evaluated like Example 32 instead of example 37 p-

ecylbenzenesulfonic acid 0,75 weight section and N.N-dioctyl amine 0.55 weight, section. The ienesulfaria-apid 0.43 weight section and hexylamine 0.23 weight section except having used 33)The resin layer was obtained and evaluated like Example 32 instead of example 38 p

34]Instract of example 39 p-tolueneauffonic-acid 0.43 weight section and hexylamine 0.23 weight iecybenzenesulfania acid 0.37 weight section and N.N-dimethyl octylamine 0.18 weight section. tion, The resin layer was obtained and evaluated like Example 32 except having used s result was shown in Table 10, ust was shown in Table 10.

35]Instead of example 40 p-toluenesulfonic-acid 0.43 weight section and hexylamine 0.23 weight becybenzenesulfonie acid 0,75 weight section and N,N-dimethyl octylamine 0.36 weight section. tion, The resin layer was obtained and evaluated like Example 32 except having used

lecylbenzanesulfonic acid 0.75 weight section and N.N-dimethyldodecyl amine 0.49 weight section. 36]instead of example 41 p-toluenesulfania-acid 0.43 weight section and hexylamine 0.23 weight tion, The resin layer was obtained and evaluated like Example 32 except having used s result was shown in Table 10.

s result was shown in Table 10.

nparative example 12 p-toluenesulfanic-acid 0.43 weight section and hexylamine 0.23 weight 37]The resin layer was obtained and evaluated like Example 32 except not having used tion. The result was shown in Table 10,

in layer was obtained and evaluated like Example 32 instead of hexyfamine 0.23 weight section 38]Comparative exemple 13 p-toluenesulfonio-acid 0.43 weight section was not used, but the 39]The resin layer was obtained and evaluated like Example 32 except not having usad ept having used decyl emine 0.36 weight section. The result was shown in Table 16.

40]The resin layer was obtained and evaluated like Example 32 instead of comparative example 15 oluenesulfania-acid 0.43 weight seation and hexylamine 0.23 weight seation except having used mparative example 14 hexylamine 0.23 weight section. The result was shown in Table 10.

41)The resin layer was obtained and eyaiusted like Example 32 instead of comparative exemple 16 oluenesulfonic-acie 0.43 weight section and hexylamine 0.23 weight section except having used lecyl–trimathylammonium—chioride 0.33 weight section. The result was shown in Table 10.

lium dodecylbenzenesulfonete 0.80 weight section. The result was shown in Table 10.

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# JP,11-343152,A [DETAILED DESORIPTION]

13   P.		メラホン器	400		34	.) 		<b>秋淡遊》。 時間</b>
P-MANDAN         P. 63         APP P         B         R         R 23           F-MANDAN         0 . 43         PP P         H         0 . 43           FPACENSAR         0 . 75         NP P         H         0 . 23           FPACENSAR         0 . 75         FAM         H         0 . 17           FPACENSAR         0 . 75         FAM         H         0 . 17           FPACENSAR         0 . 75         FAM         H         0 . 18           FPACENSAR         0 . 75         FAM         H         0 . 18           FPACENSAR         0 . 75         FAM         FAM         0 . 18           FPACENSAR         0 . 75         FAM         FAM         0 . 18           FPACENSAR         0 . 75         FAM         FAM         0 . 18           FPACENSAR         0 . 75         FAM         FAM         0 . 18           FPACENSAR         0 . 75         FAM         FAM         0 . 18           FPACENSAR         0 . 75         FAM         FAM         0 . 18           FPACENSAR         0 . 75         FAM         FAM         0 . 18           FPACENSAR         0 . 13         FAM         FAM         0 . 18		被据	表面的数	181	 ×		常和光数	(S)
F-35748298   6 - 3   3   79720   31   14   6   4   4   4   4   4   4   4   4	#4 #1	P-1927/847/8	5	424V	æ	×	75-1	2 %.
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		FF84578724598	~-	ゲンド	×	:::	φ.	e (; e
		SP34/2E/2U2/M	٠.	18173	æ	×	۶.	17.
### ### ### ### ### ### ## ## ## ### #		£73645£522457 <u>8</u>	•~	34,976	×	<u></u>	-	2 0. 1
Figure   200   2		<b>新れるでのお歌</b>	200	4554	\$649	×	102	7. 7.
		\$534634533813gg	सम्ब	4934	14.5%	14.45	:	2 8, 3
Productive		発生の物の発展	۴	43.64	ガギメ	メチル	3	~ .
1		Francy Controlling	2.4	35.75	150	4.4.2	- व्यक्	300.
9         FOW         H         H         9, 18           F-PATAGENER         0, 43         -         0         -         0           FFOW         FFOW         0, 43         -         0         -         0           FFOW         FFOW         0, 43         -         0, 33         -         0         -         0         -         0         -         0         -         -         0         -         -         0         -         -         0         -         -         0         - </td <td>50. </td> <td>ŧ</td> <td>٠</td> <td>í</td> <td>t</td> <td>ì.</td> <td>e</td> <td>7 7. 0</td>	50. 	ŧ	٠	í	t	ì.	e	7 7. 0
F-Paradath         0.43         - 0         - 0           ドランルへンセンスがおい深すをリウム         0.40         ***           ドゲンルトリメトルアンモニかるシロライド         0.33	~ v:	1	69	がいか	æ	ĸ	**	8 12 B
ドデシルペンセンスがおン談ナをガウム 0, 40 77 Fゲアルトリメナルアンモニウムラロライド 0, 88 6	*-	p-paraments		1	}	ŧ	¢3	数据光化
ドゲンルトリメチルアンモニッとうロライド 9,33 8	183 	140 July 18	など思ソス	小祭へやん	1443	.*	(	80 %: :-
	نې —	ドゲンルトリ	1×+117.	1600	. 400 G		•	89, 6

Example 32 was rinsed and dried further, and the polyviny! butyral resin which reduced the contont of sodium salt was obtained. It was 20 ppm when the sodium content of the obtained polyvinyl butyral [0243]⊺he polyvinył hutyral resin compounded in example 42 (preparation of polyvinyl butyral resin) resh was measured by 10P emission spectrometry. The particle diameter of addium salt was 3,5

[0244](Production of a resin layer) Posyvinyt-butyral-resin 100 obtained weight section, On the same layer, the moisture-proof white-blush-mark sex test was done like Example 1. The regulf, was shown weight section, dodecylbenzenasultanic acié 0.33 weight section, and the decyl amine 0.17 weight section was carried out, and the 0,8-mm-thick resin layer was obtained, About the obtained resin canditions as Example 32, it kneaded, press forming of triethylene-giyoui-di-2-ethyl-husyrate 40

dadecythenzenesustanic-acid 0.33 weight section and deayl amine 0.17 weight section except heving used dodocylbenzeneaulfonie acid 0.17 weight, sention and dacy! anine 0.09 weight section. The [0245] The resin layer was obtained and evaluated like Example 42 instead of example 43 result was shown in Table 11.

dodecylhenzenesulfonic-acid 0.33 weight section end decyl amine 0.17 weight section extent having used dodecylbenzenssulfanic soid 0.03 weight section and decyl amine 0.02 weight section. The [0246] The resin layer was obtained and evaluated like Example 42 instead of example 44 result was shown in Table 11.

evaluated like Example 42 except having used N.M-dimethyl octylamine 0.17 weight section. The 10247] Instead of example 45 decyl amine 0.17 weight section, the resin layer was obtained and result was shown in Table 11.

0248]histead of example 46 dodecy!henzenesufonio-acid 0.33 weight section and decyl amine 0.17 dodecylbenzemesulfanie avid 0.17 weight section and N.M-dimethy! octylamine 0.09 weight section. weight section, The resin layer was obtained and evaluated like Example 42 except having used The result was shown in Table 11. [0249]Instead of example 47 dodecylbenzenesulfonic–aeid 0.33 weight, section and decyl amino 0.17 dedecybenzonssuffenic acid 0.03 weight section and N.N-dimethyl octylamine 0.02 weight section. weight section. The reain layer was obtained and evaluated like Example 42 except having used The result was shown in Table 11.

dodecylbenzonosuflanic acid 0.30 weight section and N.N-dimetryldodecyl amino 0.20 weight section. [0250]Instead of exemple 48 dedecytherzeresulfonic-acid 0.33 weight saction and decyt amine 0,17 weight section. The resin layer was obtained and evaluated like Example 42 except having used The result was shown in Table 11. 2009/06/15 http://www4.ipdi.inpit.go.jp/cgi-bir/fran\_web\_cgi\_ejje?atw\_u=http%3A%2F%2Fwww4.ipdl.i..

51]Instead of example 49 dedacyBenzenesulfonio-acid 0.33 weight section and decyf amine 0.17 ght section. The resin fayer was obtained and evaluated like Example 42 except having used tecylbenzenesulfanic acid 0.12 weight section and M.N-dimethylidodecyl amine 0.08 weight section. 3 result was shown in Table 11.

52]The resin layer was obtained and eveluated like Example 42 except not having used sparative example 17 dodecylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17

ght section. The result was shown in Table 11.

53]Comparative example 18 dodecylbenzeneau/fonic-acid 0.33 weight section was not used, but reain layer was obtained and evaluated like Example 42 instead of decyl amine 0.17 weight tion except having used decyl amine 0.36 weight section. The result was shown in Table 11.

54]The resin layer was obtained and evaluated like Example 42 instead of comperative example 19 tecylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section except having addecylbenzenesulfonic acid 0.30 weight section. The result was shown in Table 11.

id godecy/Benzenesulfond acid 0.39 weight section. The result was shown in Table 11.

55] The result mayer was obtained and evaluated like Example 42 instead of comparative example 20 tecylhenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section except having of sodium dodecy/benzenesulfonate 0.50 weight section. The result was shown in Table 11, 56] The result was shown in Table 11, belylhenzenesulfonate 0.33 weight section and decyl amine 0.17 weight section except having becylhenzenesulfones and evaluated like Example 42 instead of comparative example 21 becylhenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section except having

lecylbenzenesulforbared 0.33 weight section and decyl amine 0.17 weight section except havin od dodecyl-trimethylammonium-chlorida 0.50 weight zaction. The result was slawn in Table 11, 571

57.5 bis 11]

36	~~						
	スポキン製	404		7.8	S		水炭粉5.4 時期
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رم س	ドゲン ボトロメ オツアンをこかん クロサイ	ンメチルアニ	14-31	19 ロサイ	ja.	9.8	5 4. 7

58]Potyvinyl-butyral-resin 100 weight section compounded in example 50 Example 32, On the conditions as Exemple 32, it kneaded, press forming of triethylene-glycof-di-2-ethyl-butyrate weight section, octanoic asid 0.30 weight section, and the decyl amine 0.35 weight section was ried out, and the 0.6-mm-thick resin layer was obtained. About the obtained resin layer, the isture-proof white-blush-mark sex test was done like Example 1, The result was shown in Tabla

59]instead of example 51 decyl amine 0.35 weight section, the resin layer was obtained and shated like Example 50 except having used dodecyl amine 0.40 weight section. The result was wn in Table 12.

60]Instead of example 52 decyl amine 0.35 weight eaction, the resin layer was obtaired and busted like Example 50 except having used tetradesylamine 0.45 weight section. The result was 61]The resin fayor was obtained and evaluated like Example 50 instead of example 53 octanolod 0.30 weight section and decyl amine 0.35 weight section except having used myristic acid 0.50

own in Table 12.

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JP,11-343152,A [DETAILED DESCRIPTION]

weight section and dodacyl amine 0.40 weight section. The result was shown in Table 12. [O252]Instead of example 54 decyl amine 0.35 weight section, the resin layer was obtained and evaluated like Example 50 except having used N.M-dimethyldodecyl amine 0.45 weight section. The result was shown in Table 12.

10263]The resin layer was obtained and evaluated like Example 50 instead of example 55 octanoio-acid 0.30 weight section and decyl amine 0.35 weight section except having used benzoic acid 0.30 weight section. The result was shown in Table 12. 10264]The result ayer was obtained and evaluated like Example 50 except not having used comparative example 22 decyl amine 0.35 weight section. The result was shown in Table 12. 10264]The result was shown in Table 12.

[Table 12]

		たなたン数	224		ハット	Λ		大家後24時間
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, إ	\$33 \$33	こりスチン袋	6, 50	13.64	XX	<b>~</b>	0, 40	23. 6
	U)	オクタン級	6, 39	Ť.	4. 7. 2.	ガギメ	57.14 x4.16 8, 45	29.8
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*:\$\tilde{\omega}	işit işir	オクタン教	3, 8 6	f	ı	;	0	7.8.0

60266]Polyvinyl-butyrai-resin 100 weight section prepared in example 86 Example 42. On the same conditions as Example 32, it kneaded, press forming of triethylene-glycol-di-2-ethyl-butyrate 40 weight section, Ji (n-butyl) phesphoric acid 0.16 weight section, and the dodecyl amine 0.14 weight section was carried out, and the 0.8-mm-thick resin layer was obtained. About the obtained resin layer, the moisture-proof white-blush-mark sex test was done like Example 1. After manufacturing the glass leminate like Example 1. After manufacturing the atmosphere of 50 \*\* and 80\*8H\*, an adhesive (am Meru) examination which is in Example 1 was carried out, and the adhesive property of the glass peripheral part which absorbed moisture was investigated. When the periphery had a portion (pen Meru) 0-1) with remarkable exfoliation of glass compared with the center section at this time, the width of this portion was measured and it was considered as exfoliation distance. The result was shown is Table 13.

(0267)The resin feyer was obtained and evaluated like Example 56 instead of example 57 Jl (n-butyl) phosphoric cold 0.18 weight section and dadecyl amine 0.14 weight except having used Jl (n-butyl) phosphoric cold 0.17 weight section and M.N-dänetfujl actylamine 0.13 weight section. The result was shown in Table 13.

[0268]The resin layer was obtained and evaluated like Example 56 instead of example 58 Jf (n-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used Jf (n-ethylhexyl) phosphoric acid 0.19 weight section and dodecyl amine 0.11 weight section. The result was shown in Table 13,

10269]The resin layer was obtained and evaluated like Exemple 56 instead of example 59 Jl (n-butyl) phospheric acid 0.16 weight section and dodecyl amine 0.14 weight except having used Jl (n-ethylhexyl) phosphoric acid 0.20 weight section and N.M-dimethyl octylamine 0.10 weight section. The result was shown in Table 13.

[0270]The resin layer was obtained and evaluated like Example 56 instead of axample 80 J. (n-buty) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used J. (n-dodecyl) phosphoric acid 0.20 weight section and dodecyl amine 0.10 weight section. The result was shown in Table 13.

[0271]The rasin layer was obtained and evaluated like Example 56 instead of example 51 JI (n-butyl) phosphoric ecid 0.16 weight section and dodecyl amine 0.14 weight except having used JI (2-dadecyl)

sphoric soid 0.2% weight section and M.N-dimethyl octylamine 0.09 weight section. The result was wn in Table 13.

72]The resin layer was obtained and evaluated like Example 56 instead of example 52 Jl (r-butyl) sphoric acid 0.18 weight section and dadecyl amine 0.14 weight except having used ranydphosphoric acid 0.17 weight section and dodecyl amine 0.13 weight section. The result was wn in Table 13.

73]The resin layer was obtained and evaluated like Example 56 instead of comparative example 23 n°butyl) phosphoric acid 0.16 weight section and dodacyl amino 0.14 weight except having used no (n°dodacyl) sodium phosphate 0.30 weight section. The result was shown in Table 13. 74]The result was shown in Table 13. n°butyl) phosphoric acid 0.16 weight section and dodacyl amine 0.14 weight except having used lecyl-trimethylammonium-chloride 0.33 weight section. The result was shown in Table 13.

75) 5le 13]

W889W	(40.00)	•	0	0	60	٥	5	0	50	1.1
水海德三人時間	** (%)	1 E. 5	13,3	3 2, 4	14, 4	13.4	3 5. 8	1 4. 7	52. 4	54, 7
	彩粒密数	8, 3,4	1996 1892 XFW R. 13	3, 4, 8	242 X40 2. 30	3, 8.0	X47 X48 9, 88	% %	0.80	ē. 3 3
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	ê	然出	1755	444	* PF#	21.53	4964	86.63	なかりり かかり	2640
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		85 85	;~ (5)	60 (C)	12. 12.	6.0	~ =	2 9	80 80	72

perature was held, and the reactent was deposited. Then, hold the system of reaction at 45 \*\* for sho! 275g of 98.9 mai to the preparation pure water 2390g of example 63(1) polyvinyl-acetal resin Interlayer for glass laminates, As a plasticizer. 40 copies of triethylene glycol di-2-ethyl butyrate warming --- it dissolved. Temperature control of the system of reaction was carried out to 12 \*\*. yral resin was obtained. As far the smount of residual ecety? groups, the content [ of the average id), 8.971 copy (2,8x10 "4 mol) of magnesium 2-ethylbutanoste (carbon number 6), the ultraviolat ne "lab PURASUTO mill" Oriental energy machine company) was equipped with the T die, and on 78]adding the average degree of polymerization 1700, and the saponification degrees of polyvinys re superfluous water performed rinsing and desiocation for 2 hours, and white powdered polyvinyl 773(2) As opposed to 100 popies of polyvinyl butyral resin obtained by the manufacture above of ticle diemeter of 20 ppm and neutralizing saft in sodium concentration 34-mol% 1-mol% 65-mol%. idual vinyl alcohol compenent amount was [ 1709 and degree of butyrafrizing ] 2 micrometers in hed, The sodium hydroxide salution which is a neutralizer neutralized the chloride catalyst, still iditions, the mixture obtained above was extruded, the film was produced, and the interlayer for of the weight of chloride patelysts [ 20 | g of ] and the n-butylaidehyde 148g were added, this rea of polymerization of the obtained polyvinyl butyral resin ] of neutralizing saft (NaOI) of the rye-mentioned 3GH was 100 ppm. Subsequently, the small extrusion machine (made by a trade ours, make a resotion complete, superflueus water washes, and unreacted n-butylaldehyde is boxylic soid, and stirring mixing was cerried out uniformly. The content of the organic soid in the extrusion temperature of 80–180 \*\*, and a metallic mold outlet temperature of 200 \*\* absorbent, and the antioxidant were added as metal salt (adhe sive strength regulator) of ss faminates about 0.5 mm thick was obtained.

78](3) After controlling the humidity of the interlayer for glass laminates obtained by the aufacture above of the glass laminate so that water content may be 0.4 to 0.5% of the weight in sir-conditioned room, it put between two float glasses (2.4 mm in thickness), and preliminary nesion was carried out by the rolling method. Subsequently, actual adhesion of the layered product which preliminary adhesion was carried out was carried out on condition of the temperature of 130

a.//www4.iodlampit.go.jp/ogi-bin/tran.web.ogi.ejie?etw\_o=http%3A%2F%2Fwww4.iodii...\_2009/06/15

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\*\*, and pressure 13 kg/om² in autoclava, and the glass laminate was obtained. [0279](4) The following methods estimated the pan Meru velue of the performance of the glass laminate obtained by the evaluation above. The method of the statement estimated moisture

terminate obtained by the evaluation above. The method of the statement estimated resistance in the Example 1. The result was as being shown in Table 14.

[028G]Valuation method (i) The head struck the glass laminate which was neglected for 16 hours and carried out temperature control under pan Meru value—18\*\*0.6 \*\* temperature with the harmer within the following the particle diameter of glass was set to 6 mm or feas. subsequently — following the judging standard which judges with the boundary sample which carried out grade attachment of the degree of exposure of the interlayer after glass carries out partial avulsion beforehand, and shows the result in Table 1 — as a pan Meru value — a table — the obstom, a pan Meru value — the pinary stage and (\*\*\*) 50\*\* — one menth after { - 1 and (\*\*\*) 50\*\* — it asked about three conditions two menths after. The adhesive strength of an interlayer and glass is also so large that the above—mentioned garmen value is large, and the adhesive strength of an interlayer and an extension of an interlayer and glass is also so small that a pan Meru value is small.

[0281] The interlayer for glass laminates and the glass laminate were obtained like Example 63 except having made metal sait of garboxylic acid as shown in Table 14 as Example 64 – a 69 adhesive—strength regulator contain.

[0282]in manufacture of the interlayer for comparative example 25 glass laminates, as metal sait of carboxytic acid, instead of 0.071 copy of magnesium 2-ethylbutanoate, the interlayer for glass faminates and the glass laminate were obtained like Example 63 except having added 0.04 copy (2.8x10<sup>-4</sup> mol) of megnesium scetate (carbon number 2).

2023. The interlayer for glass and the glass laminate were obtained like Example 63 except lawing made metal saft of carboxylic acid as shown in Tabla 14 as a comparative example 28 adhesive-strength regulator contain.

19284]The performance of the glass laminate obtained by Examples 54–68, and the comparative examples 25 and 26 was evaluated like Example 53. The result was as being shown in Table 14.

Table 14]

[0286]the pelyviryl hityral resin (average-degree-of-polymerization 1650 and degree % of 8.7 mol of butyral-izing.) which adjusted the combination presentation of the interlayer for example 70 glass laminates like the case of Example 65 as polyvinyl-acetal resin 32 mol of amount & and survival vinyl alcohol component amount % of residual acetyl graups of 1 mol, As the sodium content of 20 ppm, alcohol component amount % of residual acetyl graups of 1 mol, As the sodium content of 20 ppm, the thirdriver glucas of particle diameter of 2 micrometers of neutralizing saft, and a plasticizer, 38 copies of brethylarie glycal direlegated and a plasticizer, 38 copies of Example 63 except having considered it as 0.071 copy (2.8x10<sup>-4</sup> mol) of magnesium 2-ethylburaneate (carbon number 6), the ultraviolet ray absorbant and the antioxident as metal saft of carbonylic acid. The glass laminate was obtained like Example 03 using the interlayer for glass laminates obtained above.

1028/]The interlayer for glass faminates and the glass laminate were obtained like Example 70 except. having made metal salt of carboxylic acid as shown in Table 15 as Example 71 and e 72 adhesive-

### ength regulator contain.

'88]the polyvinyl butyral resin (average-degree-of-polymerization 1650 and degree % of 67 mol of yral-izing) which set sodium content of neutralizing salt (sodium chlorida) to 10 ppm by washing pure water as example 73 polyvinyl-acetal resin The interlayer for glass laminates and the glass mate were obtained like Example 70 except having used 32 mpl of amount % and survival vinyl ohal component emount % of residual acetyl groups of 1 mal.

:89]The interlayer for glass faminates and the glass faminate were obtained like Example 70 except ving made metal salt of carboxylic acid as shown in Table 15 as Example 74 – a 78 adhesiveength regulator contain.

80]In manufacture of the interlayer for comparative example 27 glass laminetes, as metal salt of inates and the glass laminate were obtained like Example 70 except having added 0.04 copy hoxylic acid, Instead of 0.071 copy of magnesium 2-ethyliutaneate, the interiayer for glass

91) The performance of the glass laminate obtained by Examples 70-78 and the comparative 3x10 <sup>-4</sup> met) of magnesium acetate (carbon number 2).

imple 27 was evaluated like Example 63. The result was as being shown in Table 15,

		***************************************	***************************************	and the state of a second second		
	0/98/84	カルボン数の会路路(接近力級後後)	5 (被收力或依条)	パンメル教	***	水浴器24時限
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~ ~	(38) (38)	7920@c93250.	0, 487 (2.3)	, j	-	2.0
24 4-	363 (88)	2-4544が数がわり 年数カルウム	0, 0 a f (3; 0) 0, 6 z f (3; 0)	70	*	130
5 14	308 (28)	2・エチギガン 旅でがわかる	5, 67 ( ( ), 8)	50	53	8
÷ ~	365 (38)	2~4年からない数でがおかる	0. 479 (2, 8)	g5 50	c.	11.7
7 0	360 (38)	S-applications	3, 087 (2, 8)	eo ac	-	e)
45 t~	300 (88)	141 (Restable	3, 698 (2, 8)	£12	~	6 24
2.2	800 (88)	sto Meritoru	9, 198 (2, 8)	60	-	3 5
56 !~	368 (88)	2-18年の数の対対が	0, 091 (2, 6) 0, 091 (2, 6)	107	8	88
bi- bi-	300 (38)	外院でわかん	8, 8 8 5 (2, 8)	361	r.	8 6

(thexanoate (4GO), The interlayer for glass laminates was obtained like Example 63 except having aviolet ray absorbent, and the antioxident as metal salt of carboxylic acid. The glass laminate was 93]the combination presentation of the interlayer far example 79 glass laminates --- as polycinyfsidered it as 0.079 copy (2.6x19 -4 mol) of magnesium 2-ethylpentanate (carbon number 7), the of butyrsf-izhrg.) 33 mol of emount % and survivel vinyl alcohol component amount % of residual tal resin --- polyvinyl butyral resin (everage-degree-of-polymerization 1720 and degree % of 66 tyl groups of 1 mol. As the sodium content of 20 ppm. 160 copies of particle diameter of 2 rometers of neutralizing salt, and a plasticizer, 39 capies of tetraethylene Glico Lod'z 2~

34] The interlayer for glass laminates and the glass laminate were obtained like Example 79 except ing made metal seit of carboxylic acid as shown in Table 16 as Example 80 – an 82 adhesiveained like Example 63 using the interlayer for glass iaminates obtained above.

36]In manufacture of the interleyer for comparative example 28 glass laminates, as metal salt of poxylic scid. Instead of 0.079 copy of magnesium 2-ethylpentaneate, the interlayer for glass inates and the glass laminate were obtained like Example 79 except having added 0.04 copy

ength regulator contain.

36]The performance of the glass laminate obtained by Examples 19-82 and the comparative mple 28 was evaluated like Example 63. The result was as being shown in Table 16. x10 -4 mol) of magnesium acetate (carbon number 2).

37] 5le 16]

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# JP.11-343152,A [DETAILED DESORPTION]

		4.23.42 4.23.42	カルボン酸の液保液(液体力線整点)	3(浓馨力缩整剂)		バンメル数	-50	大於福品本品語
		经银(添加量)	<b>(\$ 5</b> )	数加聚 (×10 'モル: 初版 络時後人 操件後目	数数	<b>黎時後</b> 人	28 KK 11	第 人 人 人 人 人 人 人 人 人 人 人 人 人 人 人 人 人 人 人
	9 2	450 (33)	3-168425248115335	\$ 78 4GB (83) 3-16863208174342 0, 078 (2, 8)	60	00	3	2.8
	3 8	4 G O (8 9)	2-25-8132 Markeda 9. 0 ?	86 480 (89) 2-2500 Markeda 9. 671 (2, 11)	42	363	8	4 6
.,,,	 	160 (35)	2-25/04/37/8875/35/3	81 160 (32) 2-25245252555 0. 087 (2, 8)	ø	3	-	2 8
100	24	例 32 468 (38)	17. (B 275 39.).	6, 985 (2, 11)	1.77	150	00	\$ 60
· LANN	90	28 158 (33)	SECTION.	0. 0 4 4 (2, A)		201	To?	54

noutralizing selt. Amount % of 1 mol of residual acety) groups, residual vinyl alcohol component amount polymerization 1659 and degree % of 68 mol of butyraf-izing.) made into 2 miorameters of particles of 31 mol \$100 copy, As a plasticizer, as metal salt of 36 copies of dinexyl ligres macharel peats (DHA), ultraviolet ray absorbent, and the antivxidant. The glass laminate was obtained like Example 83 using The interlayer for gass laminates was obtained like Example 63 except having considered it as the according to pure water considering the combination presentation of the interlayer for example 83 and carboxylic acid 0.07% copy (2.8x10 -4 mol) of magnesium 2-ethylbutanoate (carbon number 6), glass laminates as polyvinyt-acetal resin 20 ppm, the polyvinyl butyral resin (average-degree-ci-0298]The content of neutralizing salt (sodium charide) with sodium concentration by washing the interfayer for glass laminates obtained above.

(0299)The interlayer for glass laminates and the glass laminate were obtained like Example 83 except having made metas salt of carboxylic acid es shown in Table 17 as Example 84 and an 85 adhesive strength regulator contain,

(0300) in manufacture of the interleyer for comparative example 29 glass feminates, as metal salt of laminates and the glass laminate were obtained like Example 83 except having added 0.04 copy carboxylic soid, instead of 0.07% copy of magnesium 2-ethylhutanoata, the interlayer for glass (2.8x10 "4 mol) of magnesium scetate (carbon number 2).

[0301]The interlayer for glass laminates and the glass laminate were obtained like Example 83 except having made metal sait of carbuxylic acid as shown in Table 17 as a comparative exemple 30 adhesive-strength regulator contain.

(0302)The performance of the glass faminate obtained by Examples 83-65 and the comparative examples 29 and 30 was evaluated like Example 63. The result was as baing shown in Table 17,

Table 17}

2後24時期	86 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	3.8	*	2.5	2.5	ž 4
	<b>被形象33</b>	**	4	574	22	
パングラグ	经验偿人	S	LX.	0.7	un.	2
	泵	815	v	15	×	42
(旅籍力器整构)	※加黎(×10-4-4) 如郑 蔡忠康人 秦紫集 3 %	9-198032 Metebroka 6, 474 (2, 8) 3 5 3	crysters 3, 0 6 8 (2, 8) 5	2-3400000000000 0, 075 (2, 8) 5	8. 6 4 6 (2, 8)	3. 058 (2, 8)
カやボン彼の金茂岩(接着力級候権)	蒸淡	9-198892 BR173152	4247684543443	2-35年でかかれていません	数数ではは	7700000000
*****	選茲(溶加素)	% 92 BRA (36)	旅 34 DBA (38)	% SE BRA (46)	1 28 DHA (38)	CHA (SE)
		.00 .00	** **	50 (42	(4) (4)	67 67

weight section was carried out, and the O.B-nm-thick resin layer was obtained. About the obtained resin layer, the moisture-proof white-blush-mark sex test was done like Example 1. The result was [0304]Polyviny-butyral-resin 100 weight section prepared in example 86 Example 42, On the zame weight section, camphor suffonic acid 0.056 weight section, and the MN-dimethyl octylamine 0.044 conditions as Example 42, it kneeded, press forming of triethylene-glycol-di-2-ethyl-butyrate 40 shown in Table 18.

[5365]Instead of example 87 camphur sulfonic acid 0.056 weight section and N.N-dimethyl octylamine 0.044 weight section. The resin layer was obtained and evakated like Example 86 except having used hydroxypropanesulfonic acid 6.043 weight section and N.N-dimethyl octylamine 0.057 weight section. The result was shown in Table 18.

107]Instead of example 89 camphor sulfonic acid 0.056 weight section and N.M-dimethyl octylamine 44 weight section, the rezin layer was obtained and evaluated like Example 86 except flaving used decylbenzenesulforic acid 0.06 weight section and pyridine 0.02 weight section. The result was rwn in Table 18.

108]Instead of example 90 camphor sulfano acid 0.056 weight section and N.N-dimethyl ectylamine 44 weight section, the resin layer was obtained and evaluated like Example 86 except having used decylbenzenesulfanic acid 0061 weight seption and p-toluidine 0.039 weight section. The result s shown in Table 18,

09 Jinstead of example 91 camphor sufforio acid 0.056 weight section and N.N-dimethyt octyjamine 44 weight section, the resin layer was obtained and evakated like Example 86 except having used -eyclobexane 2 acetic-aoid 0.045 weight section and dodecyl amine 0.104 weight section. The ult was shown in Table 18

10]Instead of example 92 camphor sulfonic acid 0.056 weight, section and N.N-dimethy! octylamme 44 weight section, the resin layer was obtained and evaluated like Example 86 except having used laylic said 0.042 weight section and dodecyl amine 0.06 weight section. The result was shown in 3fe 18

sethyl octylamine 0.044 weight section, the resin layer was obtained and evaluated like Example 96 11]Instead of comparative example 31 camphor sulfonic sold 0.056 weight section and N.Mtent having used pyridine 0.1 weight section. The result was shown in Table 18.

edivit octylamine 0.044 weight section, the resin layer was obtained and evaluated like Exemple 86 opt having used salicylic acid 0.1 weight section. The result was shown in Table 18. 13]Instead of comparative example 33 camphor sulfonic acid 0.056 weight section and N.N-12]Instead of comparative example 32 camphor sulfonic acid 0.056 weight section and N.N-

iethyl octylamine 0.044 weight section, the resin layer was obtained and evaluated like Example 86 ethyl octylamine 0.044 weight section, the resin layer was obtained and evaluated like Exemple \$6 ept having used campher sulfane sodium 0.1 weight section. The result was shown in Table 16. 14]Instand of comparative example 34 camphor sulfonic acid 0.056 weight section and M.N-

ept having used pyridinium chloride 0.1 weight section. The result was shown in Table 18. ST.

ble 18}

29.8 2 8 3 4 6. 35. 1 88. 2 20, 5 4 3 5 19.8 28.3 34. 7 25. 3 2:54 然為當然 3044 9. 0 9866 5, 0 6 9.344 6,857 ۵, ۲ . 0 . . 0. 0 5 B | N. N. 7/9/8/9/4/7/2> 0. 0 4 3 | R. M-9/4/4/9/6/37 8. 0 5 9 3. 8-34434737 なの 4 以一 アガンゲアハン 4.0 あい しゅートグルジン 6.0 18 | 847 17 27 3 6. 08 XXXX スカカス 添加部数 4, 3 まる「物図スタセン酸ナトニウム 8 1 1,1-7,100,497 二路酸 34 機化ビリジニウム な数数 9 0 | 呼がくとれる分数 8.9 日 日かいかりまか数 8 8 37/40/26/2 58 32 キンチル数 1かリテル線 松松1247数 00: 00 61 61 ... ...

the 2-ethylbutanoic acid magnesium 0.037 weight section was carried out, and the 0.0-mm-thick ditions as Exemple 42, it knesded, press forming of N,N-dimethyl octylamine 0.11 weight, section 16]Polyvinyl-butyral-resin 100 weight saction prepared in example 93 Example 42, Triethylanetal-di-2-ethyi-butyrata 40 weight section, octanoic acid 0.4 weight section, On the same

3://www4.jpdl.inpit.go.jp/cgi-bin/tran.web\_cgi\_ejje?atw\_u=http%3A%2F%2Fwww4.jpdli.. 2008/06/15

# JP.11-343152,A [DETAILED DESCRIPTION]

resin layer was obtained. About the obtained resin layer, the moisture-proof white-blush-mark sex test was done like Example 1.

fastening the above-mentioned reain layer between the giass plates (Ax4 cm) of two sheets, and 1931 Tine friction test was done by the following method about the glass iaminate obtained by obtaining a glass laminate. The obtained result was shown in Table 19.

(9318) Friction test) After the glass laminate was immersed in underwater [with a temperature of 60 process of this underwater immersion and oven drying 3 times, the existence of exfoliation of the \*\* I for one week, it dried in oven with a temperature of 80 \*\* for 4 hours. After repeasing the interlayer in the circumference of a glass laminate was checked visually.

faminate like Example 93 was shown in Table 19 except having changed the quantity of example 94 [8318] The result obtained by performing production and evaluation of a resin layer and a glass M.N-denethyl oetylamine into 0.28 weight sections.

octanoic and into 0.1 weight sections, and having changed the quantity of N.N-dimethy; actylamine laminate like Example 93 was shown in Table 19 except having changed the quantity of example 95 19320] The result obtained by performing production and evaluation of a resin layer and a glass into 0.06 weight sections, respectively.

performing production and evaluation of a resin tayer and a glass faminate like Example 93 was shown (032) jOhange the quartity of example 98 octanoic acid into 0.2 weight sections, and the quartity of in Table 19 except having used 2-athythexanoic acid magnesium 0.045 weight section instead of 2-N.M-dimethyl octylamine is changed into 0.09 weight sections, respectively. The result obtained by ethyllutanole acid magnesium 0.037 weight section.

performing production and evaluation of a resin fayer end a glass laminate like Example 93 was shown [0322]Change the quantity of example 97 cetanoic soid into 0.1 weight sections, and the quantity of in Table 19 except having used 2-cthylhexanolo soid magnesium 0.045 weight section instead of 2-NAM-dimethyl octylamine is changed into 0.08 weight sections, respectively. The result obtained by ethylbutanoic acid magnesium 0.037 weight section.

0.037 weight section. The result obtained by performing production and evaluation of a ream layer and section instead of MM-dimethy! octylamine 0,11 weight section and 2-ethylbutanoic sold magnesium 10323]Except having changed the quantity of example 98 octanoic acid into 0.1 weight sections, and having used decyl amine 0.08 weight section and 2-sthythexanoic acid magnesium 0.045 weight a glass laminate like Example 93 was shown in Table 19,

0.02 weight sections. The result obtained by performing production and evaluation of a resin layer and 10324]Except having used di(2-ethythexyt) phosphoric acid 0.03 weight section instead of example 99 octanoic-acid 0.4 weight section, and naving changed the quentity of N.N-dimethyl octylamine into a glass laminate like Example 33 was shown in Table 19.

[0228]using the glass laminate obtained by the comparative example 35 comparative example 12, the result obtained by doing only a friction test like Example 93 was looked like [ Table 19 ], and was

aminate like Example 93 was shown in Table 19 except not having used comparative example 35 [0326]The result obtained by performing production and evaluation of a resin layer and a glass octanoic axid and M.N-dimethyl actylamine.

lanimate like Exemple 93 was shown in Table 19 except not having used comparative example 37 [D327]The result obtained by performing production and evaluation of a resin layer end a glass M.N-dimothyl octylamine.

10328]The result obtained by performing production and evaluation of a resin layer and a glass laminate like Exemple 93 was shown in Table 18 except not having used comparative example 36 potanois acid,

93 was shown in Table 19 except having used 2-ethylhexanols ecid magnesium 0.045 weight section [03.29] Comparative example 39 octannic soid and N.N.-dimethyl actylamine are not used. The result obtained by performing production and evaluation of a resin leyer and a glass laminate like Example instead of 2-ethylbutanoic acid magnesium 0,037 weight section.

0.31 weight section, and 2-ethyllurtanoic seid magnesium 0.037 weight section. The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except having used octandic acid 0.2 weight section and 2-ethylhexanoic ecid magnesium 10330 Instead of comparative example 40 octanoic-soid 0.4 weight section, N.M-dimethy; aciylamine

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#### 45 weight section.

I weight section, and 2-othylbutancic acid magnesium 0.037 weight section, The result obtained by forming production and evaluation of a resin layer and a glass laminate like Example 93 was shown Table 19 except having used octanoic soid 0.1 weight section and 2-ethylhexanoia soid magnesium 3) Instead of comparative example 41 octangic-sold 0.4 weight section, N.N-dimethyl octylamine 45 weight section.

32] ible 19]

,		····	****	·	····	·		·,	,	,	····	,	<b>,</b>	,	,
% \$\	845 2,45	*	is:		×	*	減づ	×	*	2 K	亲	火	*	統	*
水放鐵 8 4 4 %	(%) (%)	2 4. 3	2 K. S	2 2. 3	1. 25 G	20 ° 23 ° 23	3 8.	20,03	2 % &	20 20 21	3.8.	98 -1	4 b. \$	28.9	25.2
14	***	3.83.7	3.837	3.887	2000	2065	3045	2003	ſ	2633	0027	9833	8 8 8 8 8	0.045	6.045
方案数シンキンシム	% %	2-184 BR 7841	2-33を 製器 7/3/1/2	2-356 聚酸 25553	B-SERVICE METASOR	S-assendo Resissa 3. 0 d 5	かっぱんからい 飲かがらからな	P-354 鐵鐵7543.5		2-298 BBC-24304	2-198 SER 19890A	2-158 BBC194305	2-1664-83 BR284784	R-1766ARY BRUSSON G. G. G.	2-278/497 Berjansk
	<b>原创的数</b>	8	8 % .0	4. C &	ور دي دي	9. GB	ම ස ද	α α	í	;	;	 60	i	1	ı
7.1.7	<b>X</b>	X 6-29665956555	R. K-35665986725	R, R-8544575522	8, N-75542 19477.V	N. N. 55-54(9:542)	ゲケキアミン	8, N-537502354732	ł	**	ž.	B. H. PASSESSERVE	;	1	
	泰加斯黎		4	3 . 5	94 5	۵,۵	 	0.0	;	ı	, c	1	ł	0) ©	 
客級款	<b>38</b> .55	おクラン語	オクタン数	パケケン物	ギクタン製	ギクケン数	ドクタン数	\$8~ 6 (24+384×2)6	ì	}	※ハルハ※	į	:	なかかり 数	おいきのか
		99. Çı	() ()	ده ۲/2	g G	2= 120	60	8	Co O2	89 83	>	<b>60</b>	60 52	<b>Q</b>	-7°

In --- warming --- it dissolved. Temperature control of the system of resotion was carried out to 12 33]adding the average degree of polymerization 1700, and the degree's of polyvinyl alcohol 275g of of chloride catalysts [ 201 g of ] and the n-butylaldelyde 148g were added, this temperature assium hydroxide solution which is a general-purpose neutralizer neutralized the chloride oatalyst, ke a reaction complete, superfluous water washes, and unreacted n-butylaldehyde is flushed. The l also white powdered polyvinyl butyral resin was obtained through rinsing and desinnation for 2. irs with superfluous water. The amount of 64-mol % and survival acetyl groups of the degrae of anification of 98.9 mol to the preparation pure water 2990g of example 100(1) polyvinyl-acetal s held, and the reactant was deposited. Then, hold the system of resction at 45 \*\* for 3 hours, nation of average butyral of this resin was 1-mol %.

kness were obtained. It was 45 ppm as a result of messuring the kalium content of the obtained lecylbenzenesulfonic soid as organic acld, adding 0.25 copy of dimethy! octylamine as ansine and y carrying out melt kneading with a roli mill, with the press-forming machine, press forming was erage-degree-of-polymerization 1700 and amount % of 1 mei of degree % and survival acetyl sticizer, 40 copies of triethylene glycol di-2-ethyl butyrate. After having added 0.75 copy of ried out for 30 minutes, and 150 \*\* of interlayers for glass laminates of 0.76 mm of average 34/(2) As opposed to 100 copies of manufacture above-mentioned polyvinyl butyraf resin ups of formation of everage butyral of 64 mol) of the interlayer for glass laminates. As a wayer by ICP furninescence ultimate analysis.

ch exists in the interlayer for glass laminates obtained above using time-of-flight type secondary 35)it was 5 miorometers as a result of measuring the particle diameter of the potassium eloment mass spectroscopy (TOF-SIMS).

utes at 90 more \*\*. Thus, this adhesion was performed for the glass laminate by which preliminary esion was cerried out for 20 minutes on pressure 12 kg/cm² and canditions with a temperature of rubber bag and carrying out indirect desulfurization mind with the degree of vacuum of 20torf for inate with a transparent float glass (30 cm by 30 cmx2.5 mm in thickness), After putting this in in 36)(3) it inserts from both sides using the manufacture above-mentioned interlayer of a glass minutes, it moved to oven, deaersted, and the vacuum oress was earried out, holding for 30

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JP, 11~343152,A (DETAILED DESCRIPTION)

135 \*\* in air type autoclave, and the glass laminate was obtained.

8837](4) About the glass laminate obtained by the evaluation above, the humidity test was done by the following methods. The result was as being shown in Table 20.

[0336]Based on humidity test JIS R-3212 "the safety glass test method for cars", a glass faminate is neglected for two weeks under the atmosphere of 50 \*\*-95%RM.

then, the distance (white-blush-mark distance) of the mikked portion was measured from the oircumference of a glass laminate,

0.40 copy of dodecyl amine instead of 0.25 copy of direthyl ootylamine as amine. Kallum centent was 0.75 copy of dodecybenzenesulfonic acid. The interlayer for gass laminates and the glass laminate 0330]In menufacture of the interlayer for example 101 giess laminates, as organic acid, instead of were obtained like Example 100 except having added 0.30 copy of petsnoic seid and having added

(0340)th was 4 micrometers when the particle diameter of the potassium element which exists in the dodecylbenzenesulfonio acid as organic acid expept having added 0.20 copy of di(2-ethylhexyl) 10341] In manufacture of the interlayer for example 102 glass laminates, the interlayer for glass laminates and the glass laminate were obtained like Example 100 instead of 0.75 copy of interlayor for glass laminates obtained above was measured like Example 100. phosphoric acid. Kalium content was 55 ppm.

103423t was 3 micrometers when the particle diameter of the potassium element which exists in the interlayer for glass laminates obtained above was measured like Example 100

laminates and the glass laminate were obtained like Example 100 as an adhasives regulator except 10343In manufacture of the interlayer for example 103 glass laminates, the interlayer for glass having added 0.75 copy of potassium stearate. Kalium content was 350 ppm.

[0344]it was 8 micrometers when the particle diameter of the potassium element which exists in the interlayer for glass laminates obtained above was measured like Example 100.

10345]in manufacture of the interlayer for comparative example 42 glass laminates, the interlayer for giass laminates and the glass faminate were obtained like Example 100 except not having added the organic acid and amine of the dispersing agent. Kalium content was 47 ppm.

10346]it was 18 micromaters when the particle diameter of the putassium element which exists in the 03473m manufacture of the interlayer for comparative example 43 glass laminates, the interlayer for interlayer for glass laminstes obtained above was measured like Example 100

glass laminates and the glass laminate were obtained like Example 100 not having added the organic acid and amine of the dispersing agent, and except having changed washing time from 2 hours in 3 hours. Kallum content was 34 ppm.

0348]it was 12 miorometers when the particle diameter of the potassium element which exists in the 0349]The humidity test of the glass laminate obtained by Examples 101~103 and the comparative interlayer for glass laminates obtained above was measured like Example 100

examples 42 and 43 was done like Example 100. The result was as being shown in Table 20.

Table 20 [0320]

	Video physical and the second		1.4	2. No.16.		12	15. 20.70
		-				2	
		38	101	3	83	42	Ħ
	アセラール無役素数	~	**	v	~	~	*
4	双路ブケラール化像 (mo1光)	Ž,	24, U	3.28	58. 6	84.9	2. 
÷ 3	形件7セチル被数 (mo 13)	e.	ຍ ::	i. 8	G 	10 14	 
2	军均重合度	90.5	39. f	1386	907.1	1700 1700	1784
28	可置刺数(重叠系)	\$	\$	*	9	\$	¥
	かりかん数度(1998)	î.	43	Ş	ġġ	7.7	35
	タガラを発発(カル	55	**	35	900	90	Ħ
	数弦性対象後の飛床からの 音音系数(mm)	1.3	7.45 5.60	o: ~¦	2.3	60 (1)	90 94
ž	教会核定	0	٥	٥	0	х	×

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51] The glass faminate of Examples 100–103 by this invention showed the outstanding moisture istance so that clearly from Table 20. On the other hand, the glass laminate of the comparative imples 42 and 43 with the particle diameter of greater than 10 micrometers of the potassium ment with exists in the interlayer for glass laminates had bed moisture resistance.

52]adding the average degree of pelymerization 1700, and the degree% of polyvinyl alrohol 275g of confidation of 98.9 mai to the preparation pure water 2890g of example 104(1) polyvinyl-acetal in — warming — it dissolved Temperature control of the system of reaction was carried out to 12. 35% of chloride catalysts [ 201 g of ] and the re-butylaidehyde 148g were added, this temperature is held, and the reaction at 45 \*\* for 3 hours, he a reaction complete, superfluous water washes, and unreacted n-butylaidehyde is fluorabled, The illum hydroxide solution which is a general-purpose neutralizer neutralized the chloride catalyst, also white powdered polyvinyl outyral resin was obtained through rinsing and desiccation for 2 are with superfluous water. The amount of 64-mon 3 and survival acetyl groups of the degree of nation of average butyral of this resin was 1-mol %.

53/2) As opposed to 100 copies of manufacture above-montioned polyvinyl butyral resin erage-degree-of-polymerization 1700 and amount % of 1 mol of degree % and survival acetyl ups of formetion of average butyral of 64 mail of the interlayer for gass lanimates. As a sticizer, 40 copies of triethylene glycol di-2-ethyl butyrate, After having added 0.75 copy of lacybonzenesulfonic acid as organic acid, adding 0.13 copy of dimethyle octylamine as amine and y carrying out meit kneeding with a roll mill, with the press-forming machine, press forming was ried out for 30 minutes, and 150 % of interlayers for glass laminates of 0.76 mm of average knees were obtained. It was 50 ppm as a result of measuring the sodium content of the obtained ratayer by IOP luminescence ultimate analysis.

54]It was 4 micrometers as a result of measuring the particle diameter of the sodium element ch exists in the interlayer for glass laminates obtained above using time-of-flight type secondary mass spectroscopy (TOF-SIMS).

55/(3) It inserts from both sides using the manufacture above-mentioned interlayer of a glassinate with a transparent float glass (30 cm by 30 cmx). 5 mm in thickness), After putting this in in rubber bag and cerrying out indirect desuffirization mind with the degree of vacuum of 20torr for minutes, it moved to oven, deaerated, and the vacuum press was carried out, holding for 30 utes at 90 more \*\* This, this adhesion was performed for the glass faminate by which preliminary esion was cerried out for 20 minutes on pressure 12 kg/cm² and conditions with a temperature of at more type sutoclave, and the glass faminate was obtained.

56](4) About the glass laminate obtained by the evaluation above, the humidity test, was done by same method as Example 100. The result was as being shown in Table 21.

57Im manufacture of the interlayer for example 105 glass laminates, as organic acid, instead of 5 copy of dodecythenzenesulfonic ecid. The interlayer for glass laminates and the glass laminate is obtained like Example 104 except having added 0.30 copy of octancic acid and having added 5 copy of decyl amine instead of 0.10 copy of dimethyl octylamine as emine. Sodium contant was

58]tt was 5 micrometers when the perticle diameter of the sodium element which exists in the rayer for glass laminates obtained above was measured like Example 104.

69]in manufacture of the interlayer for exampic 106 glass laminetes, the interlayer for glass instas and the glass laminate were obtained like Example 104 instead of 0.75 copy of iesylbenzenesulfonio acid as crganic acid except having added 0.20 cnpy of di(2-ethyfacyt) sphoric acid. Sadium content, was 40 ppm

60]it was 2 miorameters when the particle diameter of the sodium element which exists in the mayer for glass laminates obtained above was measured like Example 100.

rrayer for glass laminates obtained above was measured like Example. No. 81]In mainifacture of the interlayer for example. 107 glass laminetes, the interlayer for glass Inatos and tho glass laminate were obtained like Example. 104 as an adhosives regulator except.

ing added 8.50 only of sodium stearate. Sodium content was 2.80 ppm. 82jit was 7 micrometers when the particle diameter of tha sodium element which exists in the risyer for glass iaminates obtained above was measured like Example 104.

33]In manufacture of the interlayer for comparative example 44 glass laminates, the interlayer for

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## JP,11-343152,A [DETAILED DESCRIPTION]

glass laminates and the glass laminate were obtained like Example 194 except not having added the organic gold and emine of the dispersing agent. Sodium contest was 51 ppm.

[0384]It was 20 micrometers when the particle dismeter of the sodium element which exists in the interlayer for glass laminates obtained above was measured like Example 104.

(0365) in menufacture of the interlayer for comparative example 45 glass laminates, the interlayer for glass laminates and the glass faminate were obtained like Example 104 not having added the organic acid and amine of the dispersing agent, and except having changed washing time from 2 hours in 3 hours. Sodium content was 30 ppm,

[03f6]it was 13 micrometers when the particle dismeter of the sodium element which exists in the interlayer for glass laminates obtained above was massured like Example 104.

10367]The fumidity test of the glass laminate obtained by Examples 105-107 and the comparative examples 44 and 45 was done fike Example 104. The result was as being shown in Table 2: [0368]

Table 21]

			**	教務第		Ž	光松松
- 1		393	§ 165	\$63 \$	191	27	: <u>\$</u>
	アセラール張波楽数	aļ,	~	*	4,		-τ
4	早均7子号一本化液 (如(4.1%)	97.0	51. 4 84.0	37.0	6.3 0	3,58	64.0
2 2	※分子や子が数 (35)のい)	e	5. D	9.	۵ ``	F. 6	175 44
*	罗瑞金合族	8225	1703	38. 18. 18. 18. 18. 18. 18. 18. 18. 18. 1	:703	3923	1750
90.	双数构造 (负税法)	\$	ŝ	*	22	ş	\$
	ナトリウム発文(の(50)	S\$	\$5	\$	280	5	8
	ナトリウム頻器(316)	**	XX.	94	۲.	82	13
35.0	対策性対策後の高移からの 当北紀後(mm)	5.4	1.4	۵ 	}	eo Hè	ਣ ਜਾਂ
33	8会共化	0	٥	0	0	×	z

[0369]The glass laminate of Examples 104–107 by this invention showed the outstanding moisture resistance so that clearly from Table 21. On the other hand, the glass laminate of the comparative examples 44 and 45 with the particle diameter of greater than 10 micrometers of the sodium element which exists in the interlayer for glass laminates had bad moisture resistance.

Effect of the Invention It consists of above-mentioned composition in this invention.

Therefore, without spoiling basic performance required for glass laminates, such as transparency, weathensbilly, an adhesive property, and penetration resistance, even when moreover placed into a humid atmosphere, the interlayer for glass faminates and glass laminate with few white blush marks of a glass laminate edge part can be provided.

[Transletion dene.]

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ages caused by the use of this translation. and INPil are not responsible for any

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\*\*\* shows the word which can not be translated.

the drawings, any words are not translated.

#### UTTEN AMENDMENT

[Written amendment] ing date May 13, Heisei 11

roument to be Amended Specification

im(s) to be Amended]Cleim

sthod of Amendment]Change

oposed Amendment]

piastioized polyvinyi polyvinylacetal resin films and said 0.3-0.8-mm-thick interlayer is immessed in aim 1)An interlayer for glass laminetes when it is an interlayer for glass laminates which consists \*\* water, wherein Hayes of 24 hours after is 50% or less.

aim 2)The interlayer for glass laminates according to claim i whose particle diameter of sodium in an interlayer is 10 micrometers or fess.

aim 3]The interlayer for glass laminetes according to claim ? or 2 whose particle diameter of fum sait in an interlayer is 5 micrometars or less.

ain 4]The interlayer for glass laminates according to claim 1, 2, or 3 whose sodium concentration

aim SIThe interlayer for glass laminates according to claim i whose particle diameter of potassium in interlayer is 50 ppm or less.

in an interlayer is 10 micrometers or less.

ain 61The interlayer for glass laminates according to claim 1 or 5 whose particle diameter of assium salt in an interlayer is 5 micrometers or less.

aim 7] The interlayer for glass laminates according to claim 1, 5, or 6 whose potassium icentration in an interlayer is 100 ppm or less,

sim 9]The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 8, or 7 which is a thing staining a compound which can form socium salt and potassism saft, and a complex.

sim JThe interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 8, or 7 which is a thing

taining arrine which is compatible in organic acid which is pempatible in resin and a plasticizer, sim 10]The interdayer for glass laminates according to claim 1, 2, 3, 4, 5, 6, 7, 8, or 9 which is a in, and a plasticizer.

og containing at kast one sort chosen from a group which consists of aikali metal sait and alkaline 1 a particle diameter of 3 micrometers or less and whose alkaline earth metal salt is a thing with a aim 11)The interlayer for glass leminates according to claim 10 whose alkali metal saft is a thing th metal salt.

uli metal saft of erganic acid of the carbon numbers 6–16 and whose alkaline earth metal salt is the sim 12]The interlayer for glass leminates according to claim 10 or 11 whose alkali metal aalt is the ticle diameter of 3 micrometers or less,

im 13]A glaza taminate characterized by making the intodayer for glass laminates according to m 1, 2, 3, 4, 5, 8, 7, 8, 9, 10, 11, or 12 come to intervene between glass of a couple at least. dine earth metal salt of organic acid of the carbon numbers 5~16.

sin 14]An interlayer for glass laminates whose particle diameter of sodium saft in an interlayer it is nterlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetal resin films, and O micrometers or less. 11/wwwfipdiinpit.go.jp/cgi-bin/tran.web.cgi.eje?atw\_u=http\$3A\$2F\$2Fwww4.jpdii... 2009/06/15

2009/06/15

http://www4.jodl.inpit.go.jp/ogi-bin/tran\_web\_ogi\_ejje?stw\_u=http%3A%2F%2F%2Fwww4.jodi.i..

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Giaim 15]An interlayer for glass laminates whose particle diameter of sodium salt in an interlayer it is an interlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetal regin films, and

Olaim 107the interlayer for glass laminates according to claim 14 or 15 whose sodium concentration in an interlayer is 50 ppm or less.

interleyer for glass laminates which consists of plasticized priyviny polyviny acetal resin films, and is Olaim 17.4n interlayer for glass laminates whose sodium concentration in an interlayer it is an 50 ppm or less,

it is an interlayer for glass faminates which consists of plasticized polyvinyl polyvinylacetal restir films. Olaim 18]An interisyer for glass faminates whose particle demoter of potessium salt in an interlayer and is 5 micrometers or less.

interlayer for glass laminates which consists of planticized polyvinyl polyvinylacutal resin films, and is Claim 19]An interlayer for glass laminates whose potassium concentration in an interlayer it is an

otterlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetal resin films, sodium particle diarreter of potessium selt in an interlayer is 5 minrometers or less, and is 100 ppm or less. Ciain 20]An interlayor for glass laminates whose potassium concentration in an interlayer it is an Claim 21]An interlayer for glass laminates whose potassium concentration in an interlayer it is an interlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetal resin films, consentration in an interlayer is 50 ppm or less, and is 100 ppm or less

Olaim 23] The interiever for glass laninates according to claim 14, 15, 16, 17, 18, 19, 20, or 21 which Olsim 22]The interlayer for glass isminates according to cleim 14, 15, 16, 17, 18, 19, 20, or 21 which is a thing containing amine which is compatible in organic acid which is compatible in resin and a is a thing containing a compound which can form sodium salt and potassaum salt, and a complex. plasticizer, resin, and a plasticizer.

Olaim 24]The interlayer for glass laminates according to claim 14, 15, 16, 17, 18, 19, 20, 21, 22, or 23 which is a thing containing at least one sort chosen from a group which consists of alkali metal sait and alkaline earth metal salt

with a perticle dismeter of 3 micrometers or less and whose alkaline carth metal salt is a thing with a Olaim 25] The interlayer for glass laminates according to claim 24 whose alkali matal sait is a thing particle diameter of 3 micrometers or less

elkali metal sait of organic gold of the carbon numbers 5-16 and whose askeline earth metal sait is the Olaim 28]The interlayer for glass laminates according to claim 24 or 25 whose alkali metal salt is the alkaline earth metal salt, of organic acid of the carbon numbers 5~16.

Giain 27]A glass laminate making claims 14, 15, 10, 17, 16, 19, 20, 21, 22, 23, 24, and 25 or an interlayer for glass laminates given in 26 coms to intervene between glass of a couple at least

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